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SUBSTITUTION OF WAX AND GREASE CLEANERS WITH BIO-DEGRADABLE SOLVENTS: PHASE I, PART I

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19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*

Methods have been tested for systematic evaluations of biodegradable solvents for aircraft parts cleaning, degreasing, and masking-wax removal processes. Screening tests have identified solvents that are biodegradable, can efficiently remove soils from aircraft parts in a dip tank, and meet the military specification requirement for corrosiveness (ANSI/ASTM) in processes used at the five Air Force Air Logistics Centers (ALCs). The database developed during Phase I, of this program identifies each process, the chemicals used, tank volumes, testing requirements, and alternative chemicals. To test solvent biodegrability, an ASTM method was modified and used to test a bench-scale activated sludge system by measuring chemical oxygen demand (COD) and adenosine triphosphate (ATP) over a 6-hour period to determine if the bacteria will reduce COD to below the limit established by the National Pollution Discharge Elimination System (NPDES). Solvents were categorized to identify any trends, and six were selected for in-depth pilot-scale testing.

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EXECUTIVE SUMMARY

Chlorinated hydrocarbons and petroleum distillates are presently used for cleaning, degreasing, and masking-wax removal at the five Air Force Air Logistics Commands (ALCs). These solvents are not treatable in the present Industrial Waste Treatment Plants (IWTPs) and when exhausted, must be shipped to landfills for disposal. In this program, the processes using these solvents have been identified through a data base that contains all the processes presently used at the ALCs. Data on specific processes include tasks, chemicals used, tank volumes, testing criteria, and alternative chemicals. About 200 chemical companies were contacted and samples obtained from them for tests as replacements for solvents currently in use.

Several methods have been developed to screen the solvents for biodegradability, cleaning efficiency, and corrosiveness. Biodegradability screening consisted of testing a bench-scale activated-sludge system and measuring chemical oxygen demand (COD) and adenosine triphosphate (ATP) over a 6-hour period to establish if the actions of the system can reduce the COD to below the limits imposed by the National Pollutant Discharge Elimination System (NPDES). This is a modification of an existing ASTM method and is being developed into a new ASTM method.

To screen candidate solvents for cleaning efficiency, the solvents were initially tested (solubility testing) to determine if they would dissolve or loosen the adhesion of the soils to metal. If effective, they were then tested for cleaning efficiency on that type of soil. Testing consisted of coating metal coupons with masking wax, oil, or grease and submerging them for a time in the solvent mixed according to the manufacturer's recommendations. The coupons were then removed from the solvent and their weight loss determined as a function of time.

Corrosion testing was performed for each of the solvents that met the criteria established for biodegradation, solubility, and cleaning efficiency. These tests, each lasting a week, followed the ANSI/ASTM F 483-77 corrosion test method (Reference 1).

Approximately 40 solvents passed the cleaning and biodegradability tests. The solvents that were applicable to all the metals were corrosion tested, and ten passed the test criteria. Six solvents (Exxon Exxate 1000, Triton Hemo-Sol, Calla 301, 3D Supreme, Orange Sol De-Solv-It, and Bio-Tek Safety Solvent) were selected for Phase II testing.

The-pilot scale testing will include: 21-day acclimation and biological evaluations using the Tinker AFB pilot test facility, evaluation of the solvent's toxicity to the culture, rinsing requirements, solvent life, enhancement processes, and compilation of human toxicity data available on the solvent.

PREFACE

This report was prepared by Idaho National Engineering Laboratory, EG&G Idaho, Inc., P.O. Box 1625 Idaho Falls, Idaho 83415 under Contract Number DE-AC07-76ID01570 for the Air Force Engineering and Services Center (AFESC), Tyndall Air Force Base, Florida 32403-6001. Mr Charles J. Carpenter was the Government technical program manager. This report summarizes work accomplished between 1 October 1987 and 30 March 1989.

This report has been reviewed by the Public Affairs Office and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nations.

This technical publication has been reviewed and is approved for publication.

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SECTION I
INTRODUCTION

A. OBJECTIVE

Solvents and cleaners are used at the Air Force Air Logistics Commands (ALCs) in aircraft maintenance for degreasing and removing wax and paint before repairing or electroplating the parts. Most of these solvents are classified as toxic. Many, such as the 1,1,1-trichloroethane used in vapor degreasing, cannot be treated in Industrial Waste Treatment Plants (IWTPs) that remove organic chemicals by biological processing. Waste from the process must be placed in drums and shipped elsewhere for disposal. Other solvents such as chlorofluorocarbon-113 (CFC-113 or Freon-113) and perchloroethylene will soon be placed under strict EPA control because of their potential ozone-depleting effects. The purpose of this program is to: (a) identify solvents for removing wax, grease, and oil that can be replaced by biodegradable solvents, (b) identify the biodegradable solvents that can be used, and (c) develop procedures for, and implement, their use.

This program has three phases: Phase I--Solvent Selection and Performance Evaluation; Phase II--Extended Performance Testing; and Phase III--Full-Scale Demonstration/Implementation of the Solvent into Industrial Processes at the ALCs.

Phase I included six tasks:

1. Identification of the industrial processes at the ALCs in which solvents/cleaners are used, the procedures for their use, and the processes following solvent use (rinsing, electroplating, etc.)

2. Development of quality assurance methods and procedures

3. Identification of the available biodegradable solvents

4. Determination of the biodegradability of these solvents

5. Evaluation of process enhancement methods

6. Screening of solvents to determine the performance of the biodegradable solvents for: (a) removing wax, oil, grease, and carbon, (b) biodegradability, and (c) corrosiveness.

This report presents the procedures and results of the above tasks.

In Phase II, extended process performance will be evaluated, including corrosion, economics, solvent life, process control, etc. It is anticipated that with the replacement solvents, there may be an increased organic load that may adversely affect biological treatment. Solvents will be evaluated on a 15- to 21-day basis using the bench field facilities located at Tinker AFB, Oklahoma. The effluents will be analyzed to ensure compliance with National Pollutant Discharge Elimination System (NPDES) requirements. During this time, testing will also include determination of the effective life of the solvents, evaluation of possible process enhancements, determination of solution test methods, and evaluation of more extensive corrosion and hydrogen embrittlement testing. Test results from this phase will be used to select one or more solvents for Phase III demonstration and process implementation.

In Phase III, the solvent and process developed in Phase II will be demonstrated and implemented in the Tinker AFB Industrial Processing Facility using Air Force aircraft parts.

B. BACKGROUND

Solvent cleaning is a surface preparation process designed to remove grease, oil, carbon, and soil from metal surfaces before further processing of the parts by electroplating, painting, or machining. Several types of solvent cleaning can be used--cold cleaning, immersion, or vapor degreasing. Cold cleaning consists of submerging the part in a tank of solvent, spraying the part with solvent, scrubbing the parts with solvent, or wiping solvent on the part. Some of the solvents used for cold cleaning and immersion include 1,1,1-trichloroethane, PD-680 (Stoddard Solvent), methylene chloride, Freon 113, and a 30% cresol 60% benzene solution.

Vapor degreasing is presently being used at Tinker AFB for removing wax from parts to be electroplated. In this process, the parts received from the parts cleaning area are dipped into the masking wax, Petrolite Amber B-Squared 175 (melting point 175°F), until a thick coating is deposited on the part. The wax is scraped off the areas that are to be electroplated and the surface of the scraped area is polished with pumice. Final surface preparation is completed by hand cleaning with 1,1,1-trichloroethane. The part is then processed through the appropriate plating line for deposition of the required metal. After electroplating and rinsing, dipping the part again into the hot wax removes most of the wax; the thin layer remaining is removed by vapor degreasing.

Vapor degreasing removes solvent-soluble soils and other entrapped soils from metals. In the vapor degreaser (Figure 1), the vapor zone is controlled by the height of the tank and a cooling water jacket. During operation, the metal (at room temperature) is dipped into the solvent's vapor. The vapor condenses on the part; soluble and insoluble soil is carried away by gravity as the solvent drains. When the part reaches the temperature of the vapor, the process is complete. One major advantage of vapor degreasing is that the part always contacts clean solvent. At Tinker AFB, 1,1,1-trichloroethane is the solvent used for this process.

The chlorinated hydrocarbons, including chlorofluorocarbon-113 (Freon-113), are preferred for this process because they have good solvency for organic materials, low heats of evaporation, are nonflammable, noncorrosive, and stable. These chlorinated hydrocarbons are not easily treated by the activated sludge system in the industrial waste treatment plant (IWTP), at Tinker AFB and they have recently fallen under scrutiny as potential hazards to the environment, workers, and the general population. The sludge (wax, dirt, oil, and grease) from the vapor degreaser is placed in drums and disposed of in a toxic waste dump.

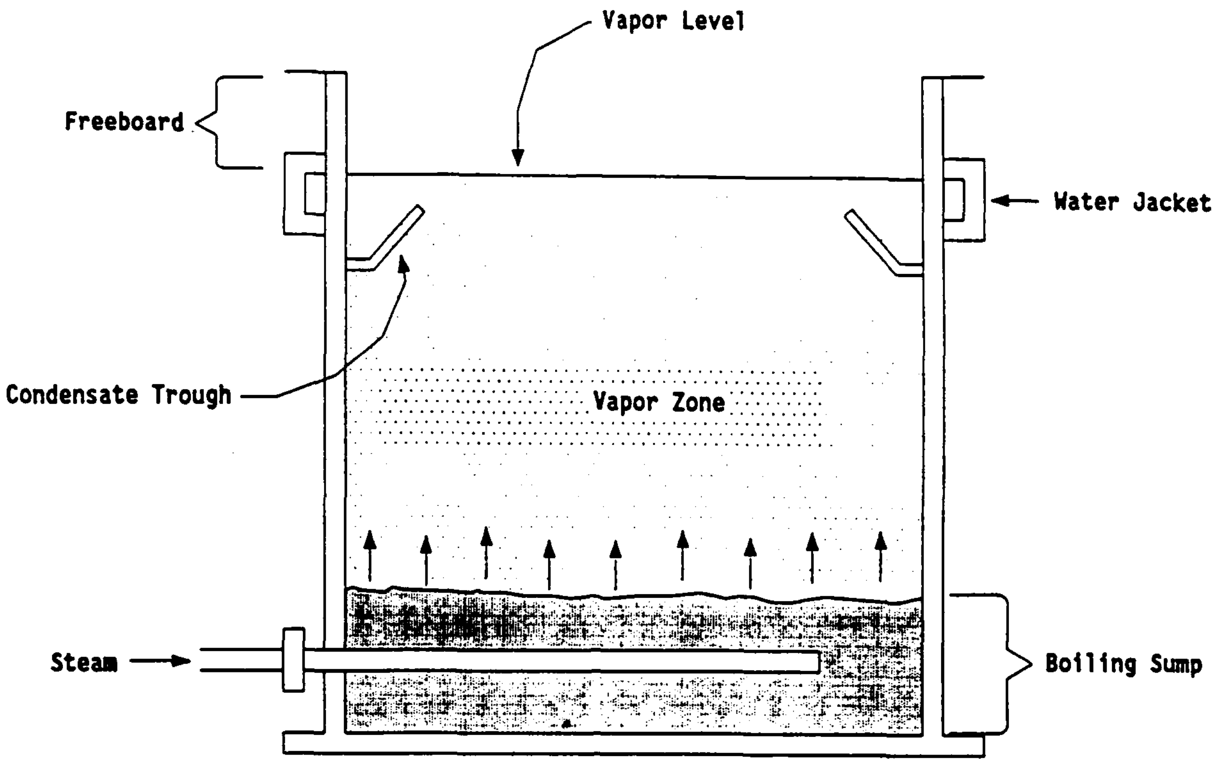

Figure 1. Schematic of a Vapor Degreaser.

Another environmental problem adding to operation costs is solvent loss by evaporation to the atmosphere, which occurs by four major routes for both cold solvent cleaning and vapor degreasing:

- Bath evaporation
- Solvent carry-out and subsequent evaporation
- Waste solvent evaporation
- Spray evaporation

Such emissions are hazardous to the operator and are potential ozone-depleting agents. Engineering controls and chemical replacement are two alternatives for controlling these emissions:

Engineering controls include placing covers on tanks, increasing freeboard ratios on vapor degreasers, developing methods to reduce solvent carry-out, and using proper operating techniques. If properly implemented, these methods can save substantial amounts of solvent, but do not alleviate the problems associated with waste sludge disposal. Also, many of these solvents will soon be strictly controlled by the EPA, which will make them more expensive and less desirable to use.

The second alternative, chemical substitution with nontoxic or biodegradable solvents is the ideal long-term solution to the present method of solvent cleaning. The substitution chemicals ultimately selected will be effective for removing greases, oils, carbon, and many other soils and may be treated in the IWTP. However, some of the solvents may require water rinsing after treatment to ensure a clean surface. In some cases, the solvents may cause oxidation on metal parts and may not clean small cracks and holes effectively due to high surface tension. These problems can be mitigated with additives to reduce surface tension and corrosion inhibitors to reduce oxidation. Some of these solvents may not be stable or be usable for as long as the chlorinated solvents.

Replacement solvents meeting the process criteria may require an enhancement process before they can clean efficiently. The processes available for enhancement can be divided into several general categories with a number of subcategories. These include:

- Immersion Cleaning. Immersion cleaning is the simplest method of cleaning. A part is simply submerged into a tank containing the solvent until the soil is removed. To enhance this process, several methods are available: (a) air agitation, consisting of air diffusers in the tank forcing air through the solvent; (b) mechanical agitation, in which a mixer increases the mass transfer of the solute into the solvent by breaking down the boundary layers; (c) tank heating to increase the solubility of the solute; and (d) ultrasonic agitation.

  Ultrasonic agitation, caused by high frequency pressure waves, causes two phenomena: microstreaming and cavitation. Microstreaming, simple eddy currents created by the standing pressure waves, works much like mechanical and air agitation, but the microstreaming cells create currents that are much more intense. High energy ultrasound can also pull apart a liquid, leaving a cavity of vapor and/or dissolved gases. When these cavities collapse, they create extremely high localized temperatures and pressures. Cavitation increases a solvent's cleaning effectiveness by introducing fresh solvent into hard-to-reach areas and by breaking soil loose. Ultrasonic agitation also greatly increases mass transfer rates. Its major drawbacks are equipment cost and operating costs.

- Spray Washers. Spray washers are available in several varieties including cabinets, conveyorized washers, and high-pressure steam washers. The basic principle of each is the same--the solvent is delivered to the part at high pressure. The high pressure breaks soil loose and forces solution into the cracks and crevices of hard-to-reach places. Cabinet sprayers employ a principle similar to that of a dishwasher--the part is cleaned in a batch mode. In conveyorized systems, a conveyor provides a continuous feed of parts to the washer.

  High-pressure steam systems are usually hand-held devices that operate in a batch mode similar to the cabinet washers, but at much higher pressures and temperatures. Cleaning solution may be added for more effectiveness, but this is required only if water alone will not do the job.

Many of these processes, including cabinet washers and high-pressure steam systems, are presently being used at the ALCs.

Evaluation of the solvents chosen for screening was divided into four subtasks:

1. Biodegradability

2. Solubility

3. Cleaning Efficiency

4. Corrosion Testing.

Figure 2 is a flow diagram of the solvent evaluation. As can be seen in the figure, each solvent was evaluated simultaneously for biodegradability, solubility, and cleaning efficiency. The method described in Reference 1 was used for the evaluations. If a solvent passed each of these evaluations, it was then tested for corrosiveness. A brief description of each of the evaluation criteria follows.

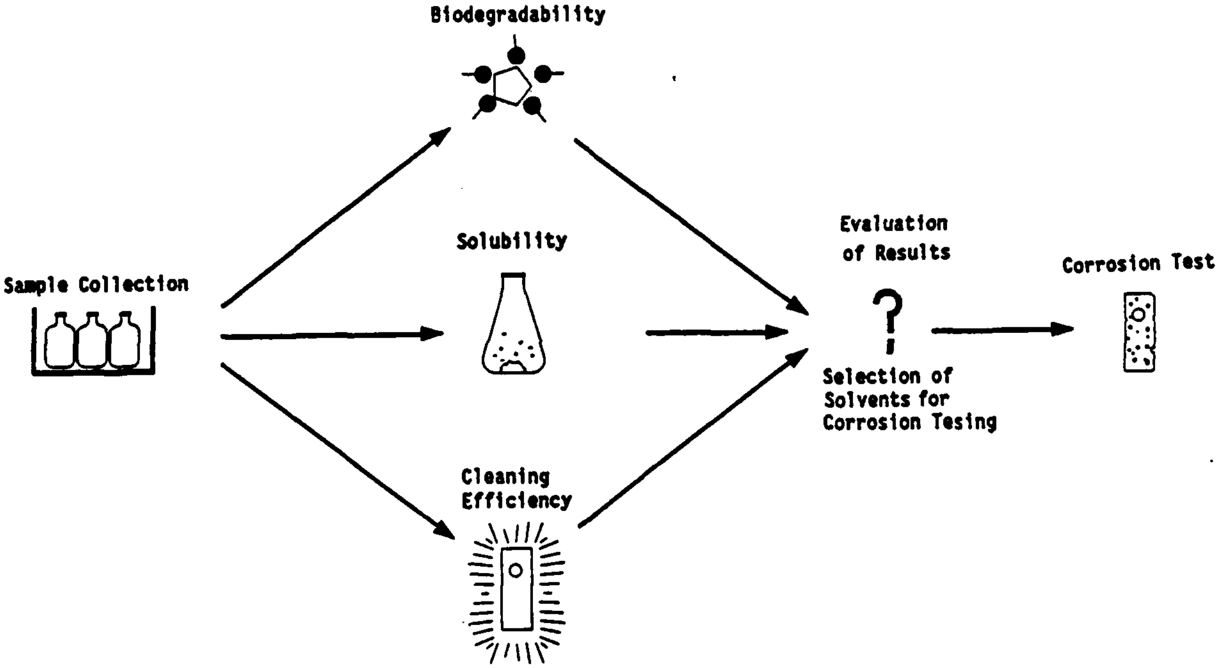

Figure 2. Flow Diagram of Solvent Evaluation Process.

- Biodegradability. A review of the literature and manufacturing information indicated that there are several methods available for determining the biodegradability of chemicals, particularly solvents, and several definitions of biodegradability. For this program, solvents that could be biologically degraded to NPDES discharge limits by the activated sludge treatment system at Tinker AFB's IWTP were considered biodegradable. A modification of ASTM's standard test method (Biodegradability of Alkylbenzene Sulfonates, Reference 2) was used to screen the biodegradability of the solvents proposed for substitution at Tinker AFB. Protocol changes were made to permit what was believed to be a more direct association of testing conditions to those conditions at the IWTP at Tinker AFB. The detailed protocol followed for this period of testing is outlined in Appendix A.

The measure of biodegradability was the ability of microorganisms to oxidize the solvent or toxic compounds in solution. This was measured as indicated by the decrease in soluble chemical oxygen demand (COD), which is a measure of the concentration of oxidized materials in the waste water that are amenable to chemical oxidation. COD is also a criterion of the Tinker AFB NPDES discharge permit (the limit is 150 mg/L COD). A culture of bacteria from Tinker AFB's activated sludge system was maintained in a bench-scale sludge column located in the Idaho National Engineering Laboratory's Idaho Research Center (IRC) in Idaho Falls, Idaho. This culture was used in biodegradation tests of candidate replacements for currently used solvents.

- Solubility Testing. Cleaning solutions work in two general ways to remove soil from a part--they can either solubilize the soil or they can break down the adhesion of the soil to the part, which allows the soil to be mechanically removed. The solubility of a solid in a liquid is determined by the intermolecular forces between the solute and solvent and also by the melting point and the enthalpy of fusion of the solute. The chemical compositions of many of the solvents we are evaluating are proprietary; therefore, the percent solubility was determined experimentally using the method described by McCoy (Reference 3).

- Cleaning Efficiency. Cleaning efficiency was evaluated by the ability of the solvent to remove wax, oil, or grease from metal coupons. A selection of eight alloys (Al2023, Al7075, Az31B, C1020, 310S, Inconel750, CDA433, and Monel K-500), representative of the alloys that are presently being vapor degreased, were used to determine cleaning efficiency. The potential replacement solvents were evaluated for cleaning efficiency as a function of time. As a baseline, 1,1,1-trichloroethane and PD-680 were used to clean the coupons. The solvents tested included both aqueous (water containing) and nonaqueous solvents.

- Corrosion Performance of Biodegradable Solvents. The purpose of this task was to determine the corrosion characteristics of the treated metals in the replacement solvents. Initial corrosion tests were the total immersion tests as recommended in ANSI/ ASTM F 483 - 77 (Reference 1). Only solvents that met the biodegradable and solubility criteria and were adequate in the initial cleaning performance tests were evaluated. The metals (Table 1) were selected from those commonly serviced at Tinker AFB. Further corrosion testing, including hydrogen embrittlement testing, will be performed in Phase II of this program.

C. SCOPE

This report presents the data from these evaluations. Included in the report is a section on methodology describing the method of collecting the information and determining the biodegradable solvents available as potential replacement solvents. Included in the methodology is the safety and health aspects of potential replacement solvents. The testing procedures, results, and conclusions are also presented.

TABLE 1. METAL SAMPLES USED FOR CORROSION TESTING

Copper, CDA110 ETP	Carbon Steel, C4340, C1020
Nickel 200	Stainless, 310S
Aluminum, AL2024	Inconel 750
Steel, C4340	Monel MK-500
Aluminum AL7075	RMI Titanium
Aluminum, AL1100	Waspaloy Alloy
Stainless, 410	Magnesium AZ31B
Admiralty Brass, CDA443	

SECTION II
METHODOLOGY

A. SOLVENT AND PROCESS IDENTIFICATION

Information on the solvents and processes currently in use was obtained by literature searches, visits to Kelly, Hill, Warner-Robins, Wright Patterson, and McClellan Air Force bases, requests to the five ALCs with Tyndall AFB support, and a tour of the Tinker AFB processes. Although a great deal of information was obtained from Tinker and Kelly Air Force bases, information is still being collected from Warner-Robins, McClellan, and Hill Air Force bases. Information requested includes processes used, tank conditions, volumes, temperatures, voltages, and chemicals.

Using Air Force Technical Orders (T.O.s) and military specifications (Mil-Specs), a computerized data base was compiled to quickly evaluate effects of replacement solvents and cleaners. The T.O. of major importance to this data system is Process Instructions Metal Surface Treatments to Meet Air Force Maintenance Requirements (T.O. 42C2-1-7) (Reference 4). This manual contains information on the aircraft maintenance processes used at the five ALCs. Each process is divided into specific tasks (cleaning, masking, plating, etc.) and explained in detail. Information presented for each task includes the chemical solution used, its composition, its preparation, procedure used to test it, process conditions, and how and where that task fits into the process as a whole. This information serves as the basic reference for the data base system.

Along with the processes information from the T.O.s, the information specific to each of the five ALCs was compiled. These data include tank numbers, volumes, and the type and supplier of the solution in the tank. The data for both Tinker and Kelly AFBs have been entered into the data base, and data are presently being compiled and entered for Hill, McClellan, and Warner Robins AFBs. In many cases, this information does not match that specified in the T.O.s; it appears that specific processes have been changed without updating the T.O. Both sets of information are, however, contained in the data base.

A menu-driven software package to support the data base allows the user to examine chemical processes in a general way or to make detailed observations of a specific tank and its chemical composition. The software guides the user from Air Force base to Air Force base, then through each process down to a given tank, its composition, and currently available alternatives as described by the T.O. manual. Figures 3-10 demonstrate the software supporting package for the data system. These figures represent the screens that appear as options are selected from the menu sets. Figure 3 shows the menu that allows selection of the desired AFB for a process investigation. For this illustration, Option 4, the first page of a list of all the electroplating processes used at Tinker AFB, is displayed on the screen (Figure 4). If "Anodize Stripping Procedure" is selected, a flow chart (Figure 5) shows all the tasks involved at Tinker AFB in this process. With the arrow keys on the computer keyboard, a specific task such as "Alkaline Clean" can be selected. Three screens are then presented: Figure 6 - the basic task information; Figure 7 - the solution information and how to prepare that solution; and Figure 8 - the frequency and method for testing each component of the solution. Figure 9 contains a compilation of the information on the process presently used at Tinker AFB. Information on alternative cleaners as specified by the T.O. is seen in Figure 10.

B. IDENTIFICATION OF BIODEGRADABLE SOLVENTS

To gather information on solvents that might be used as replacements, about 200 chemical companies, identified through the Thomas Register and personal contacts, were contacted (Appendix B). The information obtained was used to select compounds for evaluation. Appendix C contains a list of the compounds chosen for evaluation, manufacturers, and samples received.

All the information on these compounds has been entered into a data base from which the main ingredients, suggested concentrations, and any other pertinent data about the solvent or manufacturer can be referenced. Appendix D, which contains the recommended conditions at which the samples were tested, was produced from this data base.

```
A.F.B. -- TINKER

            *** SELECT AFB ***
        < 1 >  KELLY
        < 2 >  HILL
        < 3 >  McCLELLAN
        < 4 >  TINKER
        < 5 >  WARNER ROBBINS
        < 6 >  EXIT
        <Enter> SELECTION - ↑↓
```

Figure 3. Menu to Select Desired AFB for Process Investigation.

```
A.F.B. -- TINKER
PROCESS -- ANODIZE STRIPPING PROCEDURE

                          <Page Up>
    ANODIZE PROCEDURE
   >ANODIZE STRIPPING PROCEDURE
    CADMIUM ELECTRODEPOSITION ON FERROUS MATERIALS OTHER THAN LANDING GEAR
    CADMIUM ELECTRODEPOSITION ON HIGH STRENGTH STEELS (ABOVE 180,000) PSI)
    CADMIUM STRIPPING
    CADMIUM-NICKEL STRIPPING PROCEDURE
    CHROMIUM ELECTRODEPOSITION ON ALUMINUM ALLOYS
    CHROMIUM ELECTRODEPOSITION ON HIGH STRENGTH STEEL LANDING GEAR COMPONE
    CHROMIUM ELECTRODEPOSITION ON STEEL AND NICKLE AND COBALT BASE ALLOYS
    CHROMIUM ELECTRODEPOSITION ON STEEL PARTS OTHER THAN LANDING GEAR   CO
    CHROMIUM STRIPPING PROCEDURE
    CONVERSION COATING PROCEDURE
    COPPER ELECTRODEPOSITION ON CORROSION RESISTANT STEEL
    COPPER ELECTRODEPOSITION ON STEEL
    COPPER STRIPPING PROCEDURE
    ELECTROLESS NICKEL DEPOSITION ON ALUMINUM ALLOYS
    ELECTROLESS NICKEL DEPOSITION ON CORROSION RESISTANT STEEL, COBALT, AN
    ELECTROLESS NICKEL DEPOSITION ON STEEL
    ELECTROLESS NICKEL STRIPPING PROCEDURE
    INDIUM ELECTRODEPOSITION ON STEEL
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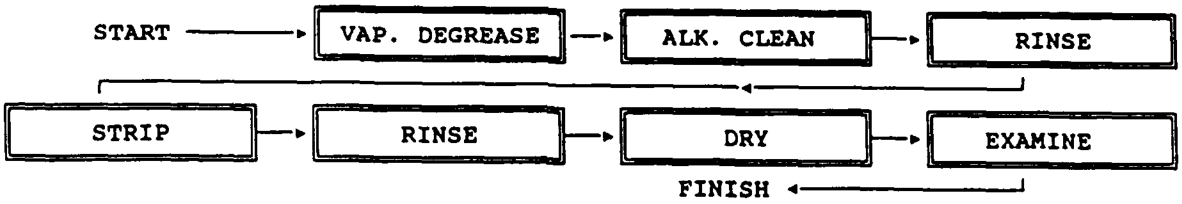
Figure 4. Processes at Tinker AFB.



<Enter> SELECTION <End> PREVIOUS MENU ↑↓→

Figure 5. Tasks of the Anodized Stripping Process at Tinker AFB.

SCREEN 1 OF 3

A.F.B. -----	TINKER		
PROCESS ----	ANODIZE STRIPPING PROCEDURE		
OPERATION --	ALKALINE CLEAN		
STEP -------	2	TIME --	1-10 MINUTES
SOLUTION ---	C-202		
CURRENT ----	IMMERSION ONLY	TEMPERATURE C --	52-57
ALTERNATE --	1		

<A> ALTERNATE
<Page Up>
<Page Down>
<End> to Exit

Figure 6. Basic Task Information of the Alkaline Clean Tank.

SCREEN 2 OF 3

ALKALINE CLEANER FOR ALUMINUM C-202

SODIUM HYDROXIDE --> 7.5 g/l (1 oz/gal) 3.8-11.3 g/l (.5-1.5 oz/gal)
TRISODIUM PHOSPHATE --> 34.5 g/l (4.6 oz/gal) 7.5-22.5 g/l (1-3 oz/gal)
SODIUM METASILICATE --> 30 g/l (4 oz/gal) 22.5-37.5 g/l (3-5 oz/gal)

1. Fill tank two-thirds full of tap water.2. Heat to 38 degrees C (100 degrees F).
3. Add materials slowly while agitating solution.
4. Fill tank to operating level with tap water.
5. Mix thoroughly.
6. Add the wetting agent.
7. Heat to 52-57 degrees C (125-135 degrees F).
8. Use continuous agitation.

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<End> to Exit

Figure 7. Solution Information for Alkaline Clean Tank.

SCREEN 3 OF 3

ALKALINE CLEANER FOR ALUMINUM C-202

CONSTITUENT	TEST FREQUENCY	TEST METHOD
SODIUM HYDROXIDE	WEEKLY	31
TRISODIUM PHOSPHATE	WEEKLY	N/A
SODIUM METASILICATE	WEEKLY	N/A

<A> ALTERNATE
<Page Up>
<Page Down>
<End> to Exit

Figure 8. Frequency and Method of Using Alkaline Clean Solutions.

AIR FORCE BASE -- TINKER
TANK NAME -- ALKALINE CLEANER

TANK NUMBER -- 100
VOLUME -- 1346

TANK COMPOSITION

SODIUM NITRATE --> 0.4 OZ/GAL
SODIUM GLUCOMATE --> 0.8 OZ/GAL
POTASSIUM HYDROXIDE --> 2 OZ/GAL
SODIUM SULFIDE --> 0.1 OZ/GAL
PH --> 12-12.5

<A> ALTERNATE
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<End> to Exit

Figure 9. Compilation of Information on the Alkaline Clean Tank at Tinker.

SCREEN 1 OF 3

A.F.B. -----	**TINKER**		
PROCESS ----	**ANODIZE STRIPPING PROCEDURE**		
OPERATION --	**ALKALINE CLEAN**		
STEP -------	**2**	**TIME --**	**1-10 MINUTES**
SOLUTION ---	**C-205**		
CURRENT ----	**IMMERSION ONLY**	**TEMPERATURE C --**	**52-57**
ALTERNATE --	**2**		

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Figure 10. Alternative Cleaners which can be used in the Alkaline Clean Tank.

One result of this program will be the protocol or methodology for selecting biodegradable solvents in the future. This will be accomplished in Phase II of this program. To develop a methodology for future screening of solvents, each solvent was categorized by the contents listed on the Material Safety Data Sheet (MSDS). The 39 categories are listed in Table 2. The categorization allows determination of the contents required for efficient cleaning and biodegradability, and is useful for screening and selecting potential replacement solvents. The information will also be useful in preparing the T.O.s for these biodegradable solvents.

C. HEALTH AND SAFETY

Four solvents are generally being used by the Air Force for soil removal and degreasing: perchloroethylene, 1,1,1-trichloroethane, trichloroethylene, and Freon 113 (CFC 113). Solvents for vapor degreasing must have several safety characteristics:

- Nonflammable and nonexplosive under normal operating conditions
- A vapor density greater than that of air
- Chemical stability (storage and/or use without degradation)
- A boiling point such that vapors can be condensed and controlled with the available cooling system
- Relatively nontoxic to operators.

These requirements limit the number of solvents available for vapor degreasing to those that are presently being used by the Air Force. Of these, CFC 113 is one of the safest from a health and safety point of view. Unfortunately, it is considered to be an ozone depleting agent and is strictly controlled by The United Nations agreement of September 1987 (the Montreal Protocol). Of the remaining solvents, several are considered to be carcinogens, and all present problems from the waste treatment standpoint.

TABLE 2. SOLVENT CATEGORIES

1. D-LIMONENE
2. PROPYLENE GLYCOL METHYL ETHER
3. DIPROPYLENE GLYCOL, MONOMETHYL ETHER
4. GLYCOL MONOBUTYL ETHER
5. ETHYLENE GLYCOL
6. GLYCOL ETHER PHOSPHATES
7. POTASSIUM PHOSPHATE
8. POTASSIUM CARBONATE, SODIUM METASILICATE
9. ETHYLENE GLYCOL MONOBUTYL ETHER
10. GLYCOL ETHER BUTYL CELLUSOLVE
11. GLYCOL ETHER, POTASSIUM HYDROXIDE
12. ISOPROPANOL
13. ACID
14. CRESYLIC ACID
15. ALKALI DETERGENT
16. ALKALINE
17. SODIUM HYDROXIDE-CAUSTIC SODA
18. AROMATIC HYDROCARBONS, DETERGENTS
19. PARAFFINS
20. SODIUM DODECYLBENZENE SULFONATE
21. BUTOXYETHANOL
22. DOWANOL DPM
23. ALKYL AMINE
24. DIETHANOLAMINE, TRIETHANOLAMINE
25. MONOETHANOLAMINE, POTASSIUM HYDROXIDE
26. PETROLEUM DISTILLATE
27. SURFACTANTS
28. ALKYL ACETATE ESTERS
29. ESTERS
30. 2-BUTOXY ETHANOL, SODIUM DODECYLBENZENE
31. TERPENES
32. D-1,8(9)-P-METHADIENE
33. PROPRIETARY
34. CYCLOHEXENE
35. NAPTHA
36. AMMONIUM

Personnel are exposed to these solvents by four main routes: bath evaporation, solvent carry-out and subsequent evaporation, waste solvent evaporation, and spray evaporation. Although engineering controls can minimize these exposures, employees can be best protected with less toxic and nontoxic solvents.

An alternative is to replace vapor degreasers with dip tanks. This process may not be as efficient, but it solves several of the safety problems associated with vapor degreasers and opens a whole range of solvents for replacement. Using liquid solvents reduces personnel exposure and decreases fire and explosion hazards.

Replacement solvents include terpenes, esters, glycols, alcohols, alkalis, and acids. Many of these have low or no flash points and LD_{50}s as low or lower than those of the presently used solvents. Table 3 compares the LD_{50} and flash points of the presently used solvents with a few of the solvents screened for replacements. From the comparison, it can be seen that the candidate replacement solvents are as safe or safer than the existing ones and offer the distinct advantage of being biodegradable. Biodegradability lowers the future risk to both the worker and the general public. Since the solvents do not have to be drummed and shipped to waste dumps, there is no chance of exposure during shipment, and no future liability. Also, the resulting reduction in long term liability will result in a cost savings.

TABLE 3. COMPARISON OF SOLVENTS' LD_{50}

	LD_{50} (oral-rat) (mg/kg)	Flash Point (°C)
Present solvents		
Trichloroethylene	4920	none
1,1,1-Trichloroethane	10300	none
Perchloroethene	none	
Freon113	43000	none
Possible Replacement Solvents		
Exxate, Alkyl Acetates	>5000	212
Desolve-it, Terpene	17750	205
Magna IV, Proprietary	>5000	none
Mirachem, Proprietary	>6000	none

SECTION III
PROCEDURE

A. INITIAL SOLUBILITY AND SOLUBILITY TEMPERATURE SCREENING

To screen the solvents quickly to determine potential solubility and solubility temperature of wax, approximately 0.5 grams of the Amber B-Squared 175 wax was placed in a test tube containing 30 milliliters of the solvent at the manufacturer's recommended concentration. The solutions were heated with a Bunsen burner while the temperature and the wax was observed. The temperature at which the wax started to go into solution was recorded as the temperature of initial solubility. It was assumed that the wax would be soluble in the solvent at a temperature 10°F greater than the initial solubility temperature.

The grease/carbon mixture was baked at 100°C for 1 hour. Approximately 0.5 gram of the mixture was placed in a test tube containing 30 milliliters of the solvent at the manufacturer's recommended concentration. The solutions were heated with a Bunsen burner to determine the temperature at which the grease starts to go into solution. These test results were used to determine the temperature to be used for the screening tests for solubility and cleaning efficiency. Due to the consistency of the carbonized oil/xylene mixture and the hydraulic fluid carbon mixture, these soils could not be screened in this manner.

B. SOLUBILITY

The solubility test was used to determine the extent that wax was soluble in the solvent tested. At the greater solubilities, the solvent was more likely to clean efficiently. Solubility was determined by McCoy's suggested method (Reference 3). A 1-gram sample of the wax was placed in a 250-milliliter round-bottom flask and 100 milliliters of solvent at the concentration recommended by the manufacturer (Appendix D) was added. The flask, fitted with a condenser, was heated for 4 hours at the temperature suggested by the manufacturers (Appendix D). The solution was filtered and the undissolved residue was dried at 105°C and weighed. From this weight the percent solubility was calculated. The standard, 1,1,1-trichlorothane, was evaluated first to establish a baseline for comparing the potential replacement solvents and cleaners. Since there was no easy means of filtering the greases and oils, they were not screened.

C. CLEANING EFFICIENCY

The solvent cleaning efficiency was determined for four different soils. The soils included Petrolyte Amber B-squared 175 wax, a carbonized oil/xylene mixture, a hydraulic fluid/carbon mixture, and a molybdenum sulfide/carbon mixture.

At present, the Air Force Bases uses the water break method to determine if a part is clean. This is basically an observation method and relies on the experience of the operator for its accuracy. It was believed that if a fluorescent dye could be used, evaluation of the parts considered to be clean would be more consistent and the method would be quick. In evaluation of the method, a yellow wax media fluorescent dye was mixed with the wax. Initial fluorescence intensity of the coupons with the yellow wax fluorescent dye mixture was 1.2-1.3%. After cleaning, the fluorescent intensity was 0.3-0.5% despite wax removals from 15-90%. Several fluorescent dyes and pigments were tested, including fluorescein and rhodamine. The yellow wax fluorescent dye mixture gave the highest initial intensity reading. However, the change in intensity did not correlate with the weight of the wax removed; intensity change was greater than the wax removed. The solvent was selectively removing the fluorescent dye from the wax.

Colormetric methods were evaluated by dissolving Sudan IV (a dye used for staining fatty tissue) in the wax to make up a 1 percent solution. Weighed samples of the wax and dye mixture were dissolved in 1,1,1-trichloroethane. Absorbance on the UV/visible wavelength of the solutions, as indicated by a spectrophotometer, could be measured from less than 100 ppb to 10 mg/L of the dye. Coupons were dipped in the hot wax and dye mixture. The weight of the mixture on the coupons ranged from 0.013 0.016 grams. The Sudan IV concentration, when the coupons were cleaned in

25 milliliters of trichloroethane was 2.3-2.6 mg/L. However, after cleaning tests with the replacement solvents, the concentration of the Sudan IV remaining on the coupons (the wax dye mixture remaining was removed with 25 milliliters of trichloroethane) showed the coupons to be 90% clean, while weight loss data showed the coupons to be only 20% clean. Apparently, the solvents selectively leach the dyes, whether Sudan IV or fluorescent, from the wax, indicating higher cleaning efficiency than actual. It was therefore concluded that the most accurate method to measure cleaning efficiency for this project was the change in the weight of the soil on the coupon.

The Amber B-Squared 175 wax (melting point 170°F) is used at Tinker AFB for masking areas of the parts before stripping and/or electroplating. To coat the metal coupons, the wax was heated to 200°F, the coupons were dipped in the wax (using the coupon dipper shown in Figure 11) and held there until they reached the temperature of the wax, and then removed slowly, so that a thin, even coating of wax remained on the

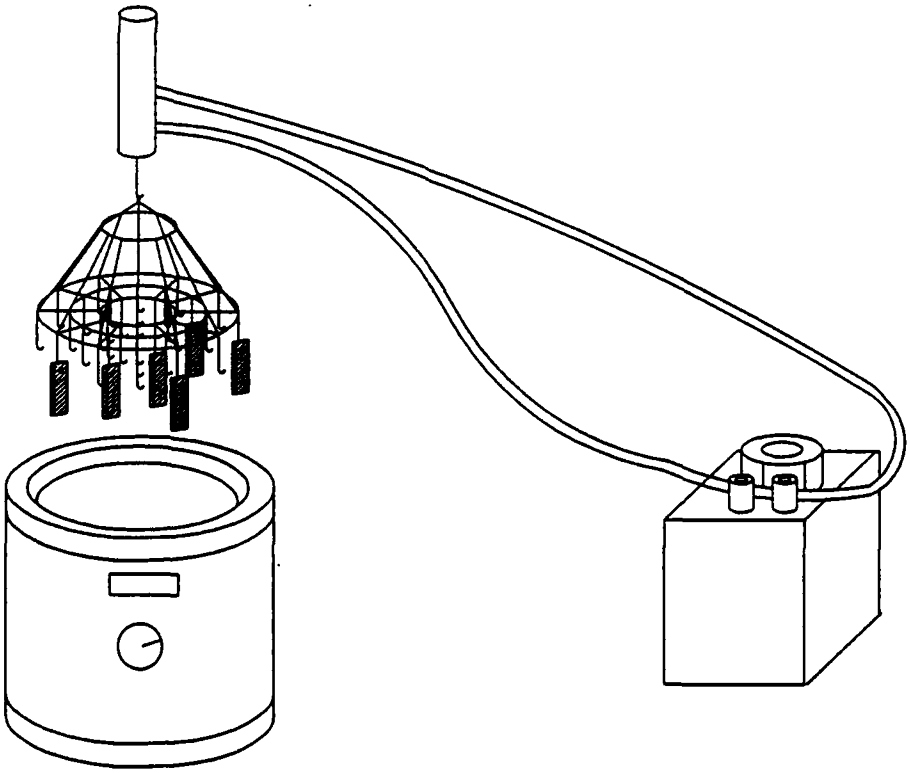

Figure 11. Coupon Dipper.

coupon. The coating, weighing approximately 0.02 gram on a 1/2 inch by 2 inch coupon, was representative of the film thickness that is normally cleaned from an aircraft part in 1,1,1-trichloroethane vapor degreasing.

The grease used was MIL-G-21164 (Reference 5) from Bel-Ray Company, Inc. Five hundred grams of the molybdenum sulfide grease were mixed with 50 grams of carbon black according to MIL-C-85570B (Reference 6). The preweighed coupons were painted with the grease/carbon mixture, covered with a tissue, and rolled with a 2-liter Nalgene bottle filled with water. The tissue was removed and the coupons were brushed to remove any excess grease and oil. The coupons were then heated at 100°C for 1 hour, cooled, weighed, and dipped in the solvents. The coupons were tested within 4 hours of preparation.

The coupons were prepared with the hydraulic fluid mixture in the same manner. Five hundred grams of hydraulic fluid MIL-H-83282 (Reference 7) were mixed with 50 grams of carbon black according to MIL-C-855708 (Reference 6). In order to have a sufficient layer of hydraulic fluid on the coupon for significant difference in weight, 2 inch by 1 inch coupons were used instead of the 2 inch by 1/2 inch.

The oil used for testing oil removal efficiency of solvents (MIL-C-43616B Mobil Oil Aero Red Band SAE50, Reference 8) had to be concentrated to 30 percent of the original weight before use; however, the procedure was not available in the MIL Spec. According to Mr. Clark, with the Department of the Navy,* the oil is oxidized by heating approximately 1 inch of the oil on a hot plate at a temperature at which the oil vaporizes only slightly. Oxidation requires 2 - 4 days, depending on the temperature and the type of oil. Approximately 475 grams of the oil were heated in this manner at 400-450°F for 14 days, after which 58 percent of the original weight remained. The consistency of the oil was like thick tar with a crisp layer on top. This was mixed with xylene and colloidal silica according to the proportions described in Mil-C-43616B (Reference 8) for the screening

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* Mr. Kenneth Clark, (215-441-1508), Department of the Navy, Warrendale, Pennsylvania.

tests for oil removal: 276 grams of xylene and 5.5 grams of colloidal silica were added to the concentrated oil and blended with a blender. The oil/xylene/silica mixture was painted onto preweighed metal coupons. The painted coupons were baked for 1 hour at 105°C, cooled, and weighed before testing. The coupons were tested within 4 hours of preparation.

The coupons were reused for subsequent solvent testing; however, the wax was thoroughly removed from the coupons beforehand by dipping in 1,1,1-trichloroethane at 66°C and rinsing with methyl ethyl ketone. Grease and hydraulic fluid were removed by wiping the coupons with a paper towel and then scrubbing with a soft-bristle brush and liquid Lava soap. The coupons were then rinsed with running water, rinsed with acetone, dried in a desiccator, and reweighed. The oil/xylene mixture remaining on the coupons after testing was removed by soaking the coupons in 30% cresol 60% Benzene for about 10 minutes and then placing the coupons and cresol/benzene mixture in a laboratory ultrasonic cleaner for 10 minutes. The coupons were removed and rinsed with acetone before drying in a desiccator and reweighing. If any oxide films were present on the coupons, they were cleaned by the ANSI/ASTM 483-77 (Reference 1) acid cleaning procedure for the respective metal.

D. CORROSION TESTING

Fifteen metals were corrosion tested in each solvent that met the biodegradability and the cleaning criteria. The metals are listed in Table 1. Corrosion testing was performed according to ANSI/ASTM 483-77 (Reference 1). The new metal coupons were cleaned by dipping in 1,1,1-trichloroethane maintained at 65°C and wiped with paper towel to remove excess solvent. They were then dipped in methyl ethyl ketone at room temperature, wiped dry, and dried in a vacuum desiccator for 15 minutes. The coupons were weighed to the nearest 0.1 milligram and hung on the corrosion apparatus for testing. Only those metals for which the solvents are recommended were tested. The samples were tested in the apparatus shown in Figure 12. The apparatus consists of a 400-milliliter straight-sided beaker fitted with a Teflon lid and coupon hanger. The system was

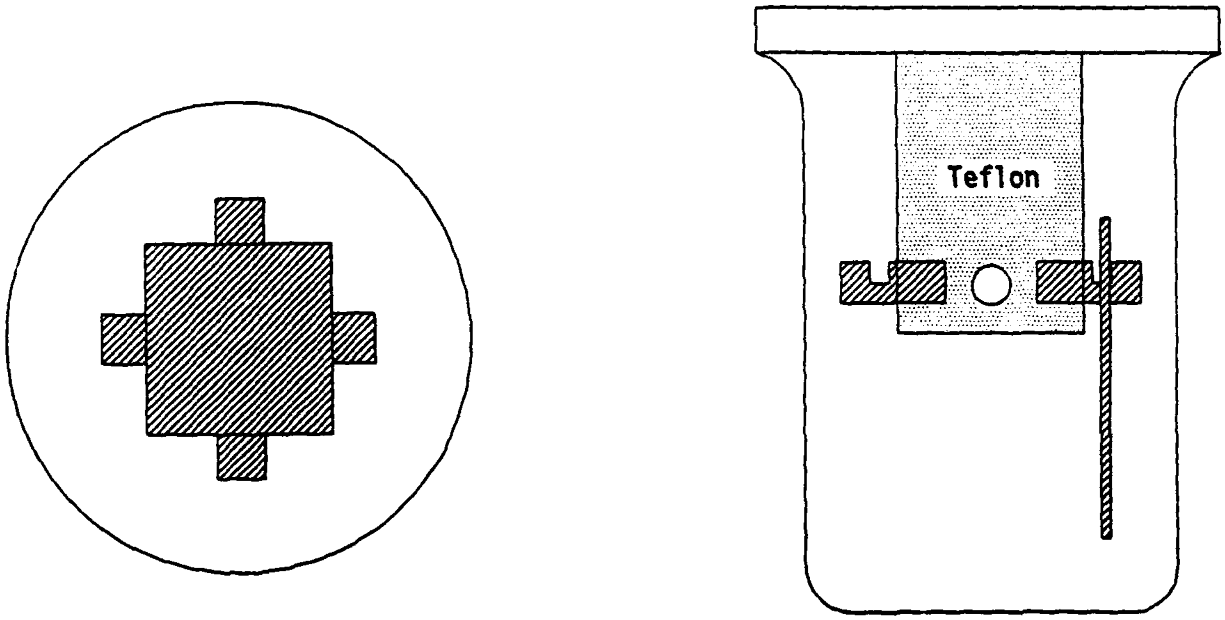

Figure 12. The Corrosion Test Apparatus.

temperature controlled with a circulating water bath. According to the test procedure, the metals were tested for 168 hours in the concentrated solvent and again in the solvent at the recommended concentration and temperature. After 168 hours, the coupons were cleaned by scrubbing with a soft bristle brush under running hot water, rinsed with distilled water, and then rinsed with acetone. The excess acetone was removed by wiping with a paper towel and the coupon was dried for 15 minutes in a vacuum dessicator. The coupons were then weighed. If any scale was present on the coupons, they were cleaned according to the acid cleaning procedure for the respective metal (Reference 1) and then reweighed. The appearance of the coupons was recorded after removing from the solvent, after scrubbing under water, and again after acid cleaning. The cleaned coupons were examined under a microscope at 480 X for pits and surface attack. Three coupons of each metal were tested in each solvent.

E. BIODEGRADABILITY

To evaluate biodegradability, 12 small columns (Figure 13) were fabricated. These columns use air diffusion for solids suspension and have sample ports which closely represent those of the actual treatment system. Samples of each solvent were mixed to recommended concentrations (as

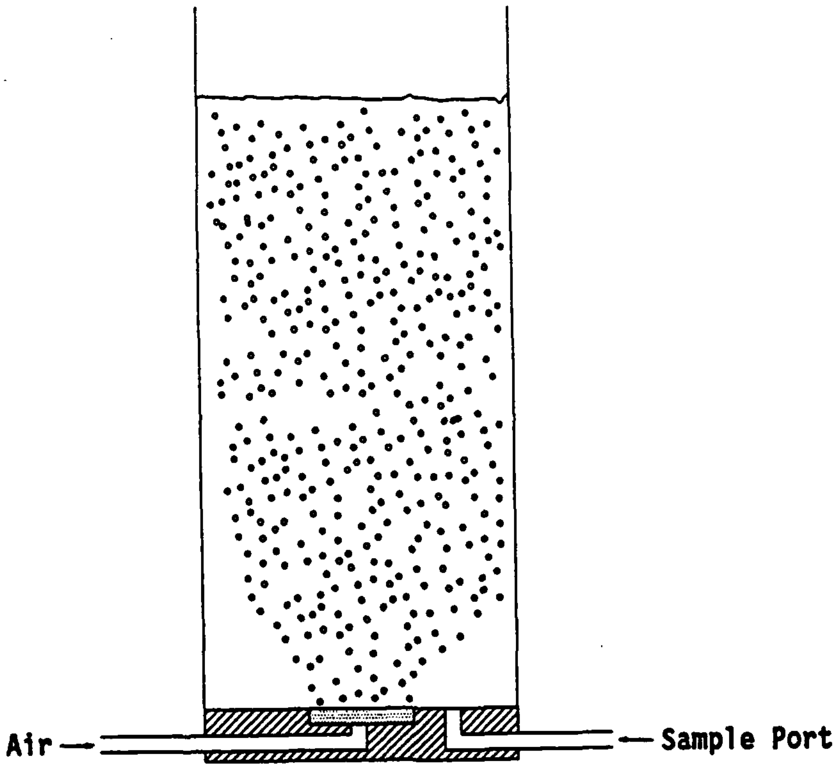

Figure 13. Biodegradability Test Column.

prescribed by the manufacturer) and then diluted 1/600 with the nutrient medium described in Appendix A. This dilution represents the concentrations that might be expected at the IWTP. The test columns were filled to a total volume of 250 milliliters; 225 milliliters of solvent and basic nutrient medium and 25 milliliters of culture-column microorganisms (The microorganisms were from a culture taken from Tinker AFB's activated sludge system.) A phenol control culture was used to compare the COD of the solvent to that of phenol. For each test column, two samples were taken every hour for 6 hours. A split, unfiltered sample was also taken during each test to determine if the solvent was being adsorbed on the biomass. COD was determined for each sample and plotted against time. An adenosine triphosphate (ATP) measure of each column was also taken at the beginning and end of each test to determine if the solvents changed the biomass. ATP was determined with a Turner Design ATP Photometer and the Internal Standard Procedure provided with the instruments.

The solvents were analyzed for COD and total organic carbon (TOC) before any biodegradation of the solvent. The TOC provides a measure of comparison to indicate the proportion of the COD that can be attributed to the organic carbons present in the solvents. TOC was analyzed using the O. I. Corporation Total Carbon Analyzer and the direct injection procedure provided with the instrument. COD was measured using the HACH COD reactors and the HACH prepackaged reagents. The concentrations were read with the HACH DR3000 spectrophotometer. To ensure a consistent correlation of biomass to solvent/sample ratio, the dry weight of the activated sludge was determined for each set of tests; the mixed liquor suspended solids (settling rate) were also determined.

SECTION IV
RESULTS

The results of the solubility, biodegradability, cleaning efficiency, and corrosion tests are presented in this section.

A. SOLUBILITY SCREENING

As an indication of the required solubility and cleaning temperatures, tests were run to determine the temperature at which the wax and grease starts to go into solution. The results for the wax are listed in Appendix E. Wax solubility tests were run at temperatures 5-10°F greater than the initial dissolution temperature (solubilities are also listed in Appendix F). For the most part, these temperatures indicated the temperature at which the wax was soluble and also was indicative of the temperature at which the solvent would remove the wax efficiently from the coupons. It was found that unless the solubility of the wax in the solvent was greater than 70%, good cleaning efficiency could not be achieved. Solubility testing could not be performed on the grease/carbon mixture, the carbonized oil/xylene mixture, or the hydraulic fluid/carbon mixture since there was no way to filter. However, the initial point of dissolution temperature of the grease/carbon mixture was not indicative of the temperature at which good cleaning efficiency could be achieved. It is assumed that stirring during heating dispersed the grease rather than dissolved it.

B. CLEANING EFFICIENCY

Coupons of a number of different metals were coated with the soils and then cleaned in potential replacement solvents. A baseline for 1,1,1-trichloroethane was determined by vaporizing 100 milliliters of the solvent in the bottom of a 300-milliliter tall beaker and hanging 10 soiled coupons in the vapor layer. In addition, cleaning efficiencies were determined by immersing the coupons in the baseline solvents. Included were 1,1,1,-trichloroethane at ambient temperature, PD-680 at ambient temperature, and the cresol benzene mixture at 140°F. The coupons were removed and treated in the same manner as the cleaning test procedures for the replacement solvents. Figure 14, 15, and 16 show the plot of cleaning efficiency as a function of time for the respective baseline solvent and soil. The coupons were 96% clean (96% of the wax was removed) after 5 minutes exposure and nearly 100% clean after 10 minute exposure to the 1,1,1-trichloroethane vapor. There was little difference in the cleaning efficiency if the coupons were rinsed or left unrinsed. The hydraulic fluid carbon soil was removed to the same extent.

Approximately 86% of the grease/carbon soil and oil/xylene soil, was removed in ten minutes in 1,1,1,-trichloroethane at ambient temperature. At ambient conditions the wax was only 75% removed. However, the other soils were removed to the same extent as with the solvent in the vapor form. PD-680 removed 95% of the hydraulic fluid, 95% of the carbonized oil/xylene, and 84% of the grease, but only 55% of the wax in 10 minutes at ambient conditions. In the 67% benzene/33% cresol solution at 140°F, all four soils were removed to nearly 100% in 10 minutes.

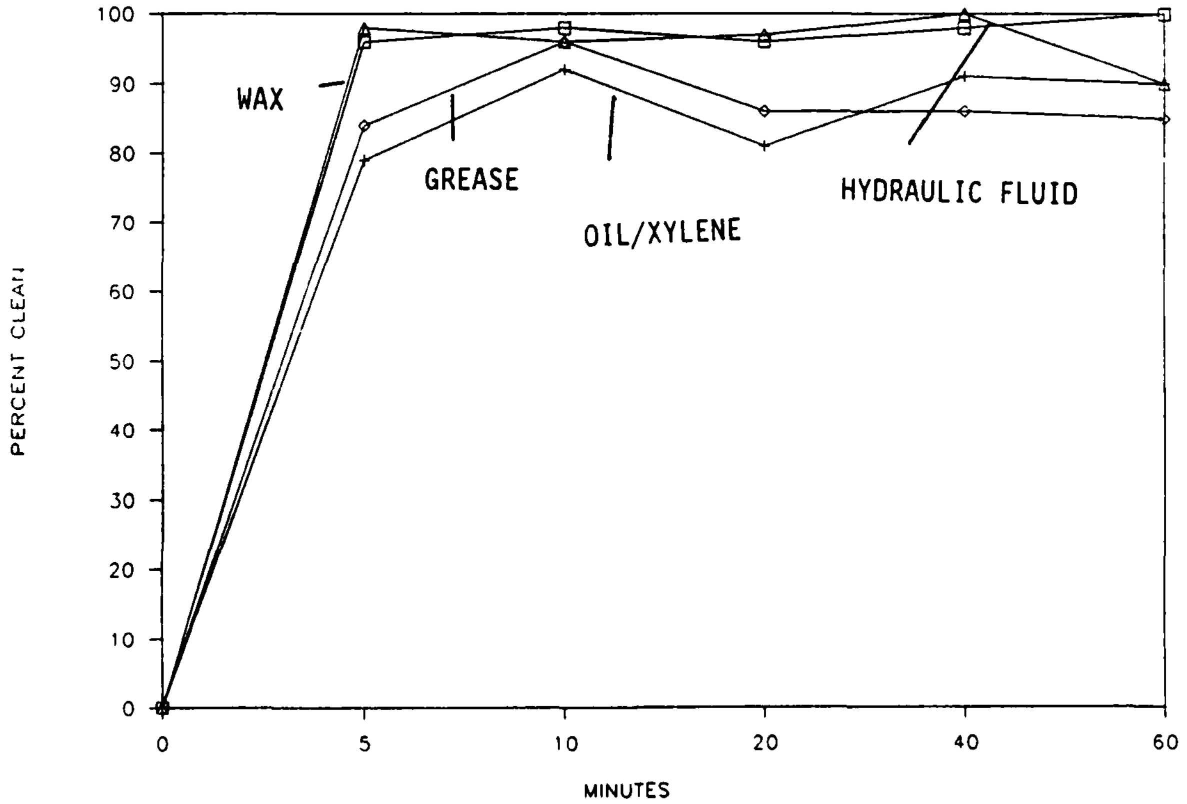

Figure 14. Cleaning Efficiency with 1,1,1-Trichloroethane.

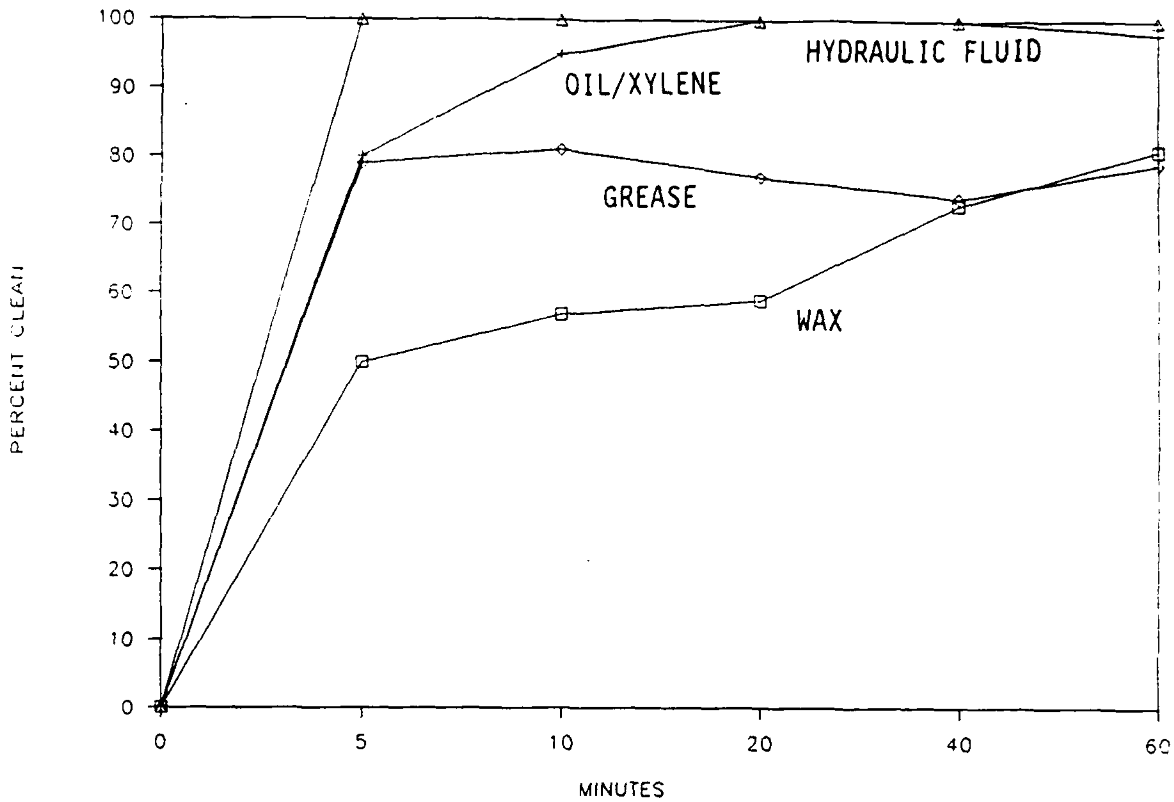

Figure 15. Cleaning Efficiency with PD-680.

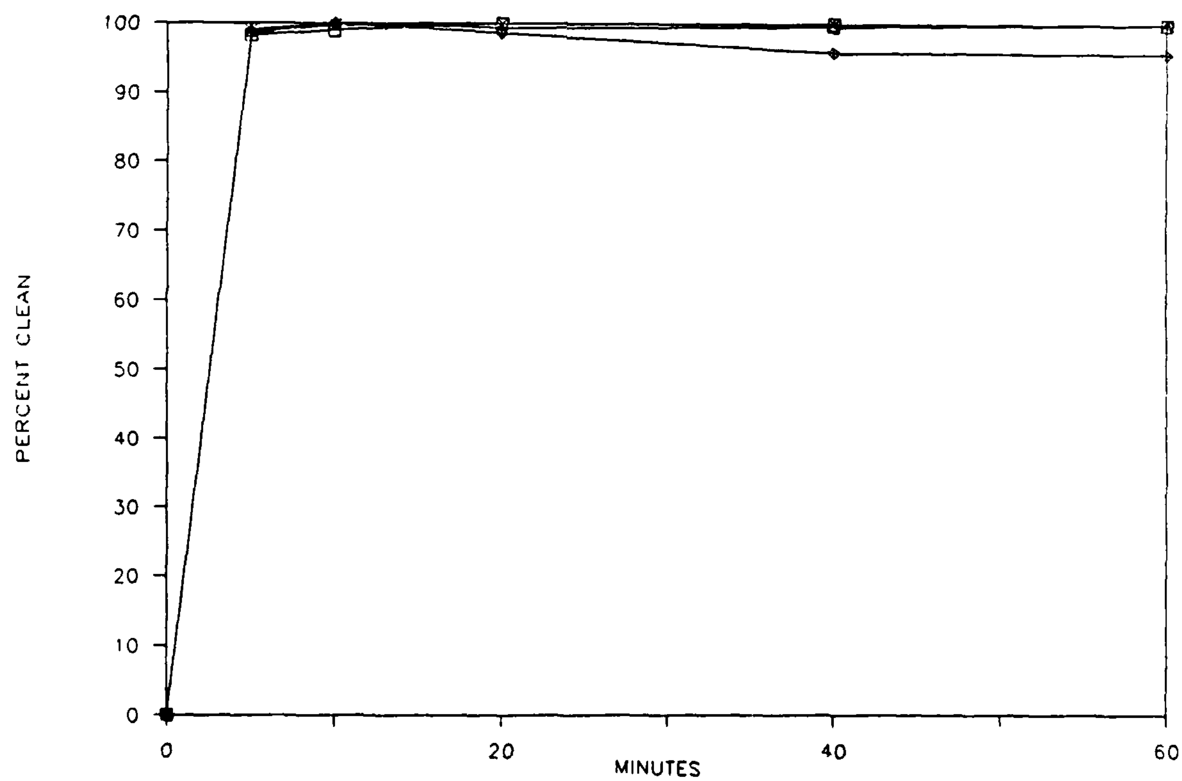

Figure 16. Cleaning Efficiency of the Benzene/Cresol Mixture for Grease, Wax, Hydraulic Fluid, and Oil/Xylene.

Another baseline was determined by dipping the soiled coupons in 200 milliliters of 1,1,1-trichloroethane at ambient temperature, PD-680 at ambient temperature, and 67% benzene with 33% cresol at 55°C. These are representative of the solvents used in immersion and cold cleaning.

Comparison tests were run to determine if the metal composition affects the cleaning capacity of the solvent. Exxon Exxate 1000 concentrate at 100°F was the solvent used for the comparison. Figure 17 is a graph of cleaning efficiency as a function of time for some of the metals used in the cleaning tests. Although there is some scatter in the data, there is less than 10% difference in cleaning efficiency for the different metals. Any difference in cleaning efficiency should be due to the difference in solvent; therefore, the solvents' cleaning efficiency was not tested with all the metals. Appendix G contains the cleaning efficiency graphs of all the solvents tested and all the soils.

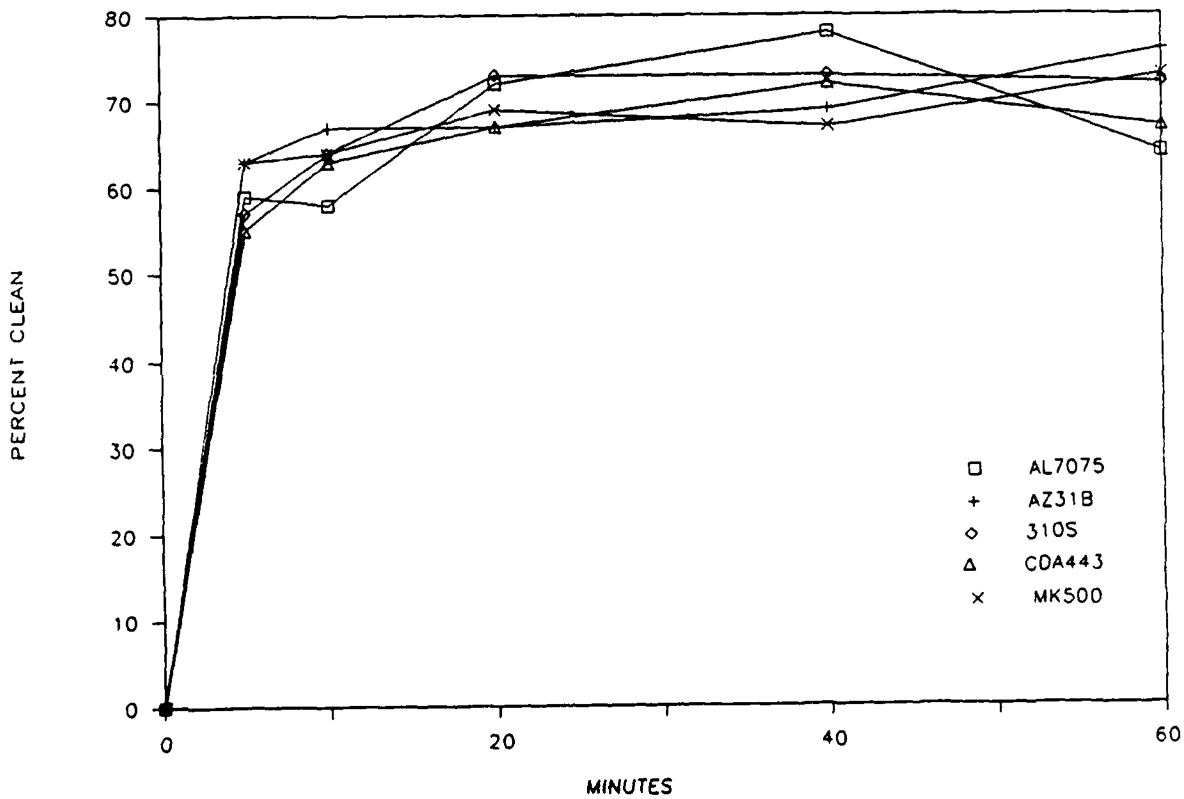

Figure 17. Cleaning Efficiency of Exxon 1000 on Different Alloys.

Grease and hydraulic fluid were much easier to remove than the wax, as shown by the comparison in Figure 18. Even after baking for one hour at 105°C, these soils could, for the most part, be wiped off the coupons. The carbonized oil/xylene mixture formed a hard glassy layer on the coupon and in many cases was as difficult to remove as the wax; however, in some cases, this soil removed more easily than the wax. A comparison of the wax, grease, oil/xylene, and hydraulic fluid is shown in Figure 19. The difference in soil removal depended on the solvent. For Orange-Sol Desolv-It at 140°F, the four soils were removed in the same manner from the unrinsed coupons. However, as shown in Figure 20 for 3D Supreme, little or no oil/xylene was removed from the coupons with 3D Supreme at 100°F while 100% of the hydraulic fluid was removed. But at 160°F (Figure 21), the oil/xylene mixture was almost completely removed while the wax was not.

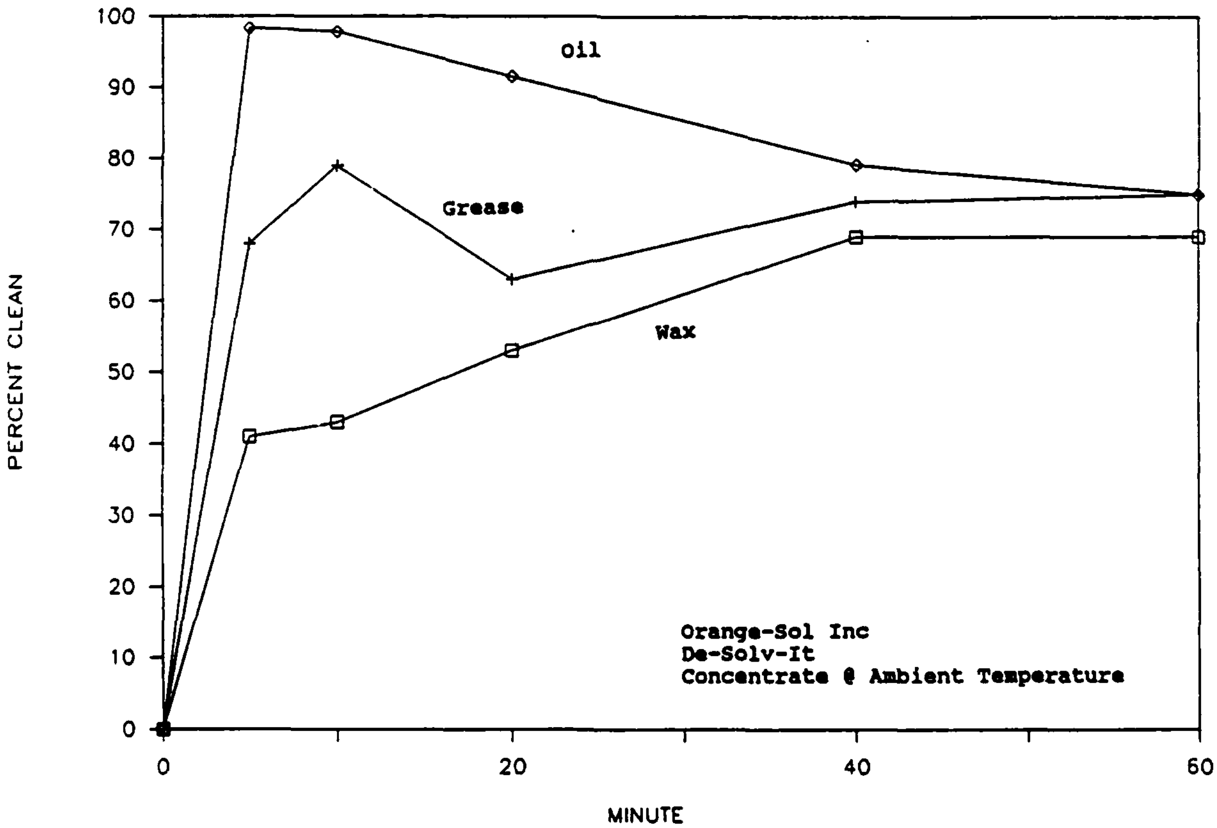

Figure 18. A Comparison of the Ease of Cleaning with Wax, Grease, and Oil.

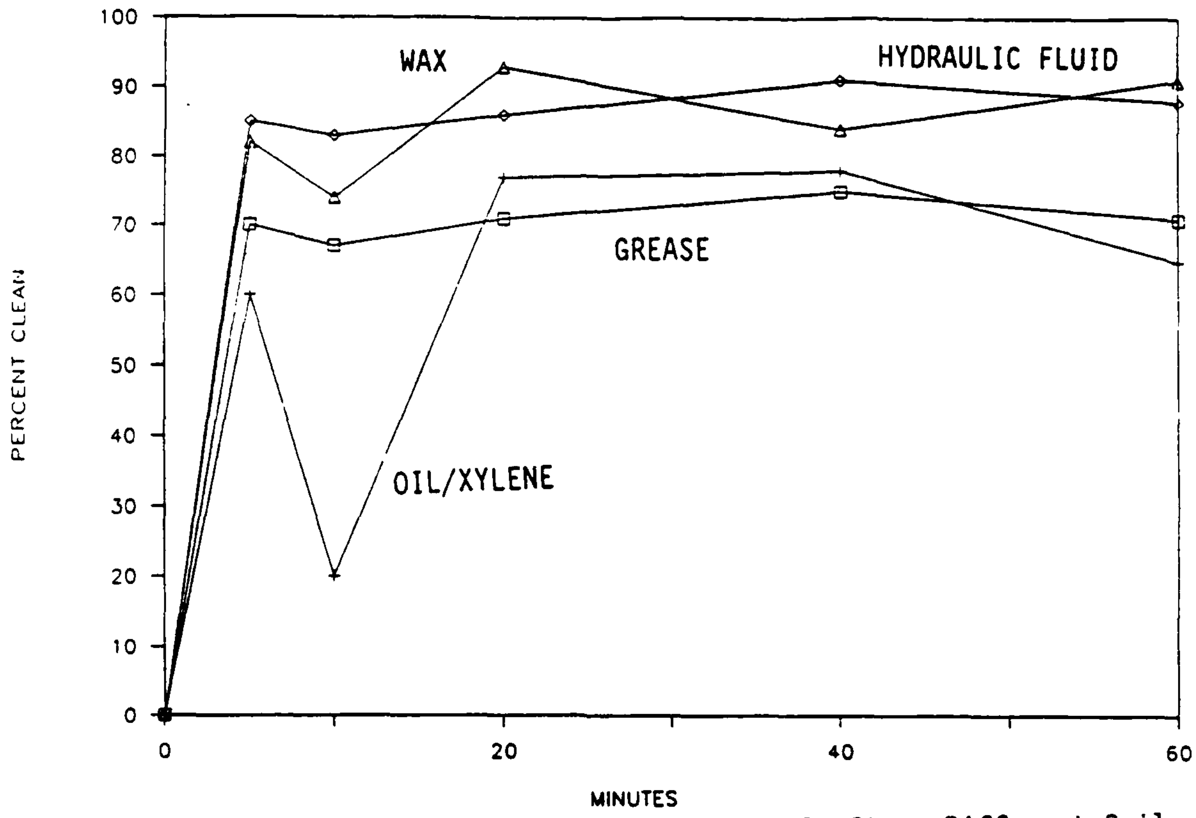

Figure 19. Cleaning Efficiency of Orange Sol De-Solv-It on Different Soils at 140°F.

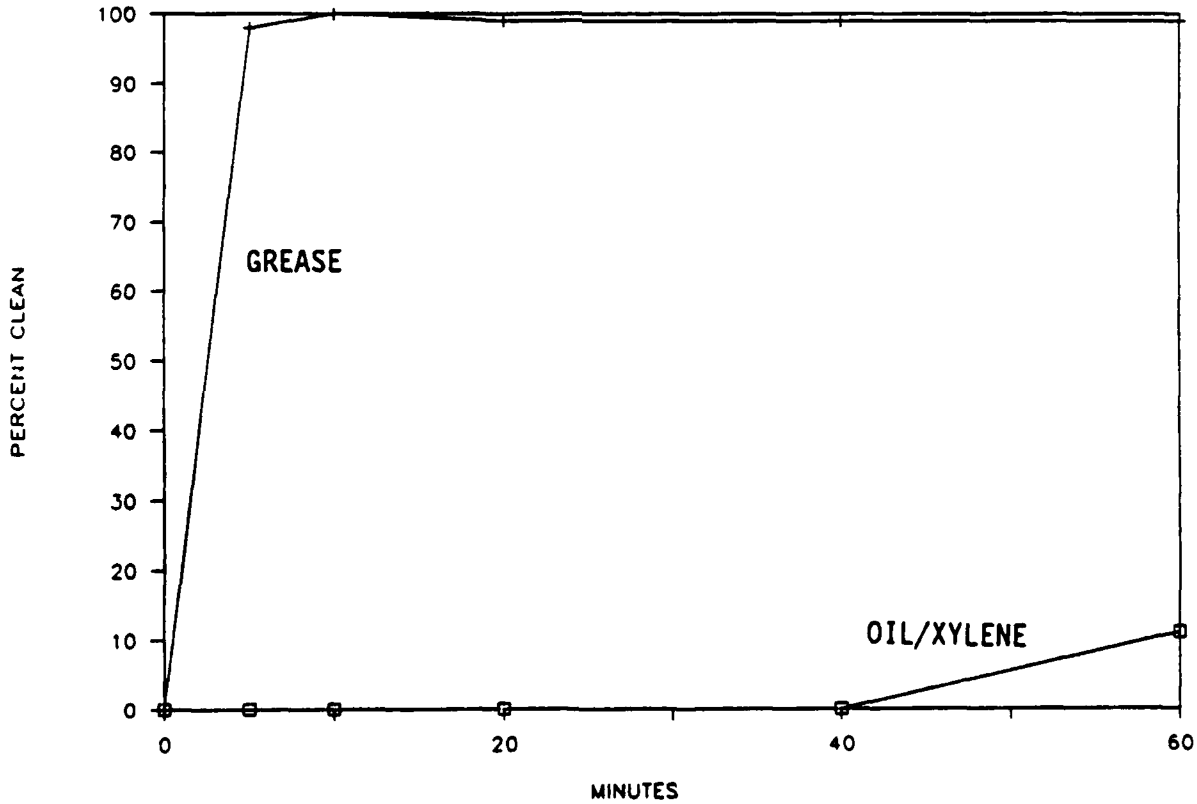

Figure 20. Comparison of Cleaning Efficiencies of 3D Supreme on Oil/Xylene and Grease.

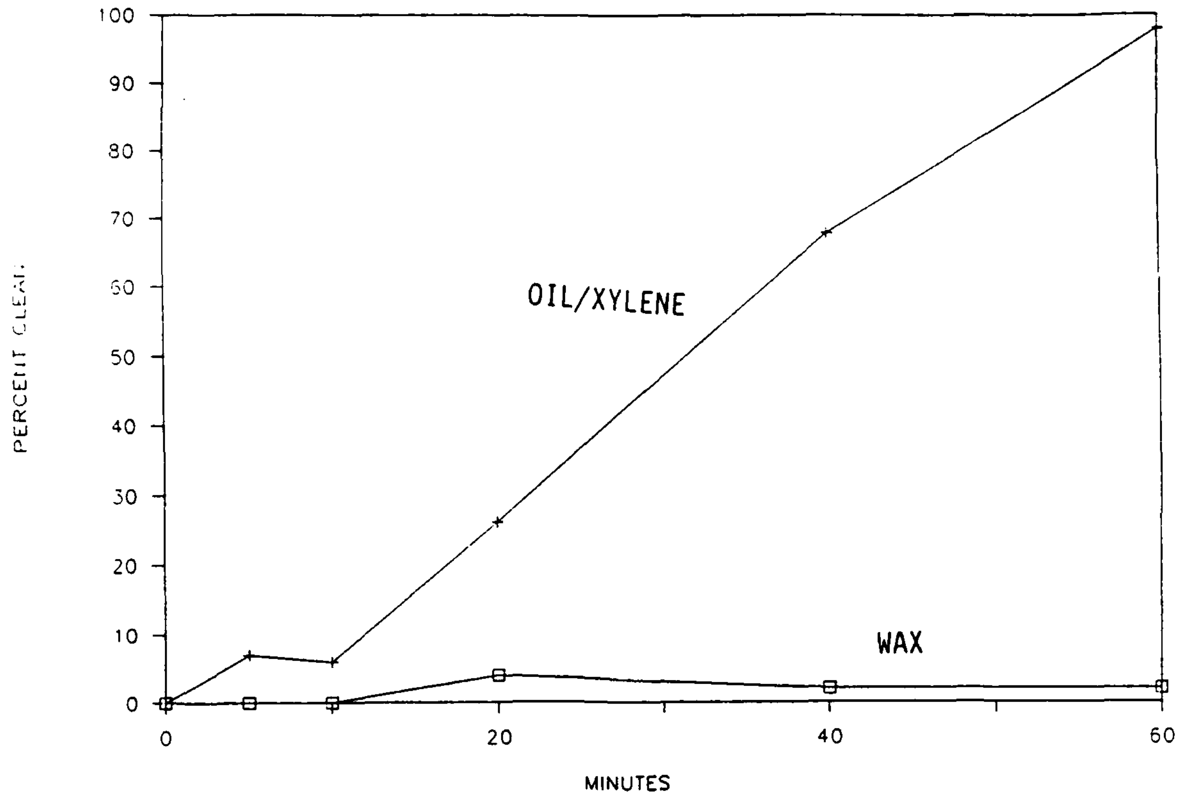

Figure 21. Cleaning Efficiency of 3D Supreme on Oil/Xylene and Wax at 160°F.

To define clean, or to select solvents for further testing, a criterion of 80% removal (in the case of the wax and oil/xylene) in 10 minutes was established. In addition, if more than 60% removal occurred in 10 minutes, the solvent was selected as one for which cleaning could be improved by one of the enhancement process. Table 4 lists the solvents whose cleaning could potentially be enhanced. Several of these solvents were tested at the temperature at which enhancement could be recommended by stirring the solution with a magnetic stirrer. In some cases (Figure 22) stirring made a significant difference in cleaning efficiency, increasing the efficiency to greater than 80% clean in 10 minutes. In other cases, there was little difference, even with stirring (Figure 23).

In a comparison of the solvents; cleaning efficiency, three different curves occurred if cleaning efficiency was plotted as a function of time. Examples of these are shown (Figures 24, 25, and 26) for grease, Pacific Chemical Riddall at 1:4 concentration and ambient temperature, wax in

TABLE 4. SOLVENTS REQUIRING ENHANCEMENT

Company	Product	Conditions
3D	Citrigold	Concentrate at 180°F
3D	Citrigold	1:1 at 140°F
Bio-Tek	134 Hi-Solv	Concentrate at 140°F
Brulin	Nature-Sol	Concentrate at Ambient
Brulin	Nature-Sol	Concentrate at Ambient With stirring
Crowley	Methyl Napthalene 5	Concentrate at 100°F
Exxon	Exxate 1000	Concentrate at 100°F
Exxon	Exxate 1300	Concentrate at 120°F
Exxon	Exxate 600	Concentrate at 100°F
Exxon	Exxate 600	Concentrate at 120°F
Exxon	Exxate 700	Concentrate at 100°F
Exxon	Exxate 800	Concentrate at 100°F
Exxon	Exxate 800	Concentrate at 120°F
Exxon	Exxate 800	Concentrate at 100°F With stirring
Exxon	Exxate 900	Concentrate at 120°F
Orange	Desolv-It	Concentrate at AMBIENT
Orange	Desolv-It	Concentrate at 120°F
Orange	Desolv-It	Concentrate at 140°F
Zep	Big Orange	Concentrate at Ambient

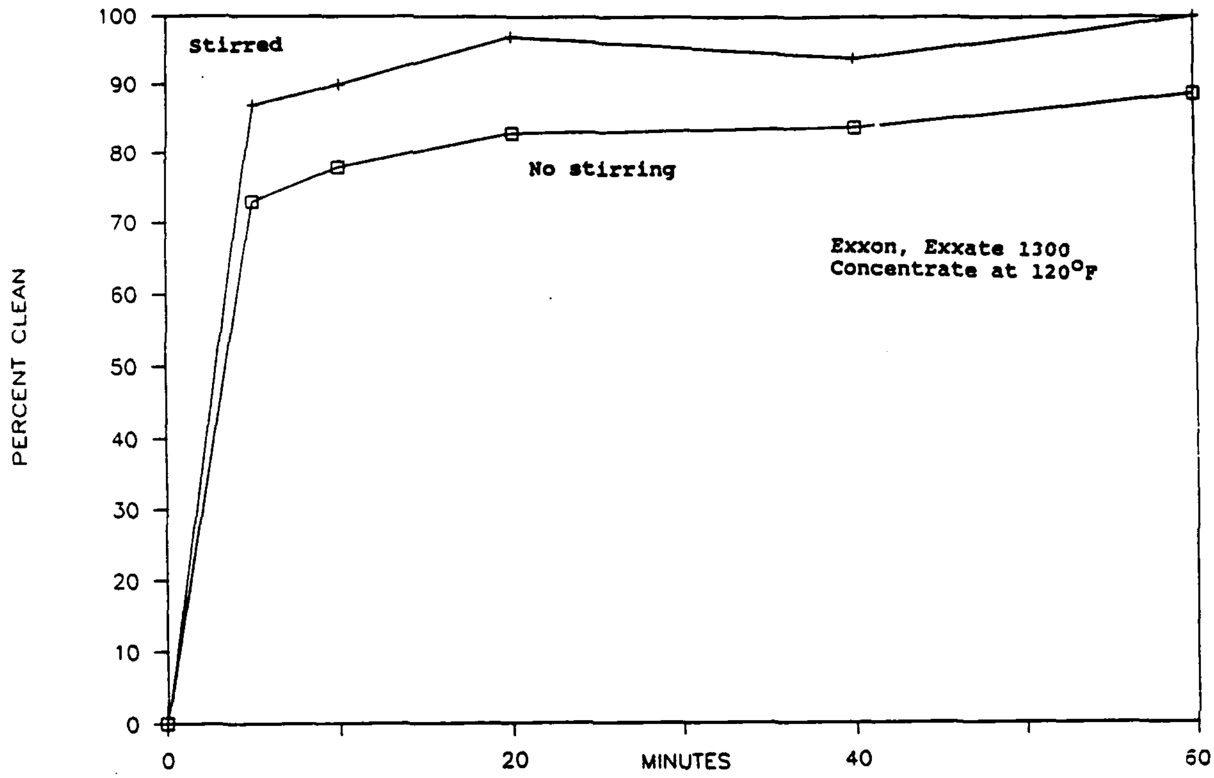

Figure 22. Cleaning Efficiency Aided by Stirring.

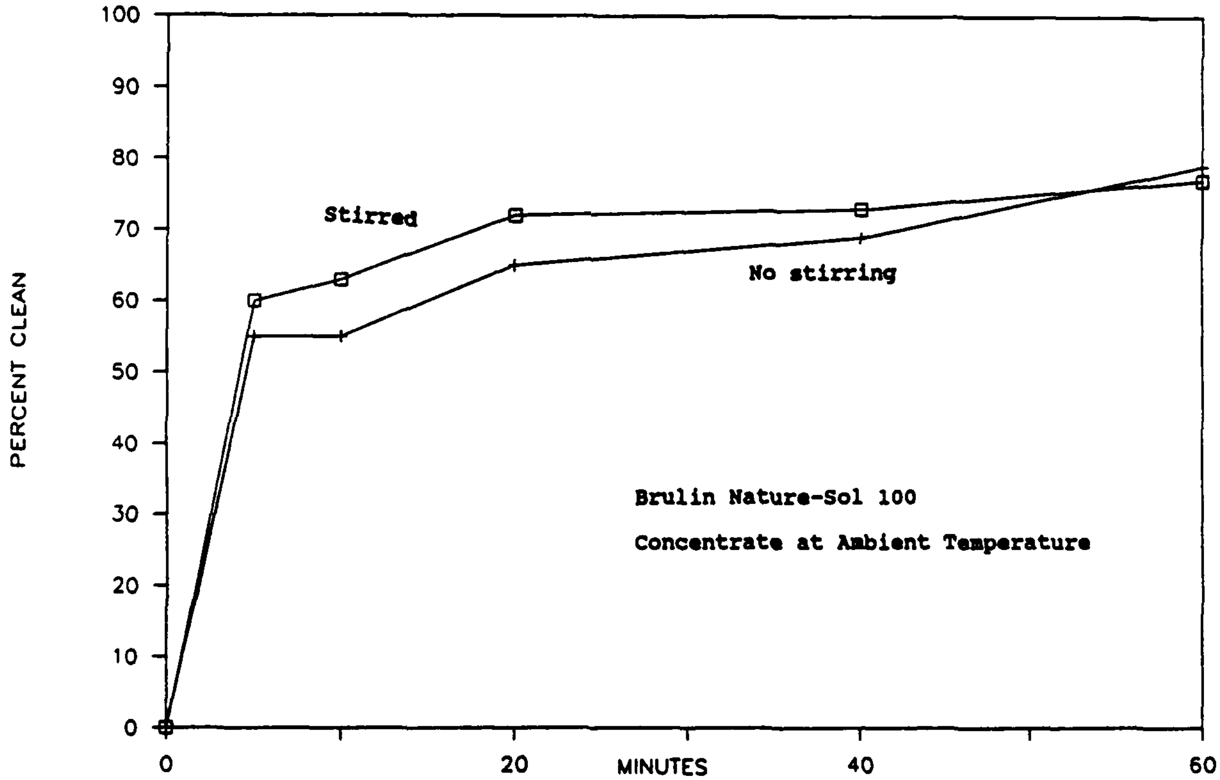

Figure 23. Cleaning Efficiency when Stirring Had No Effect.

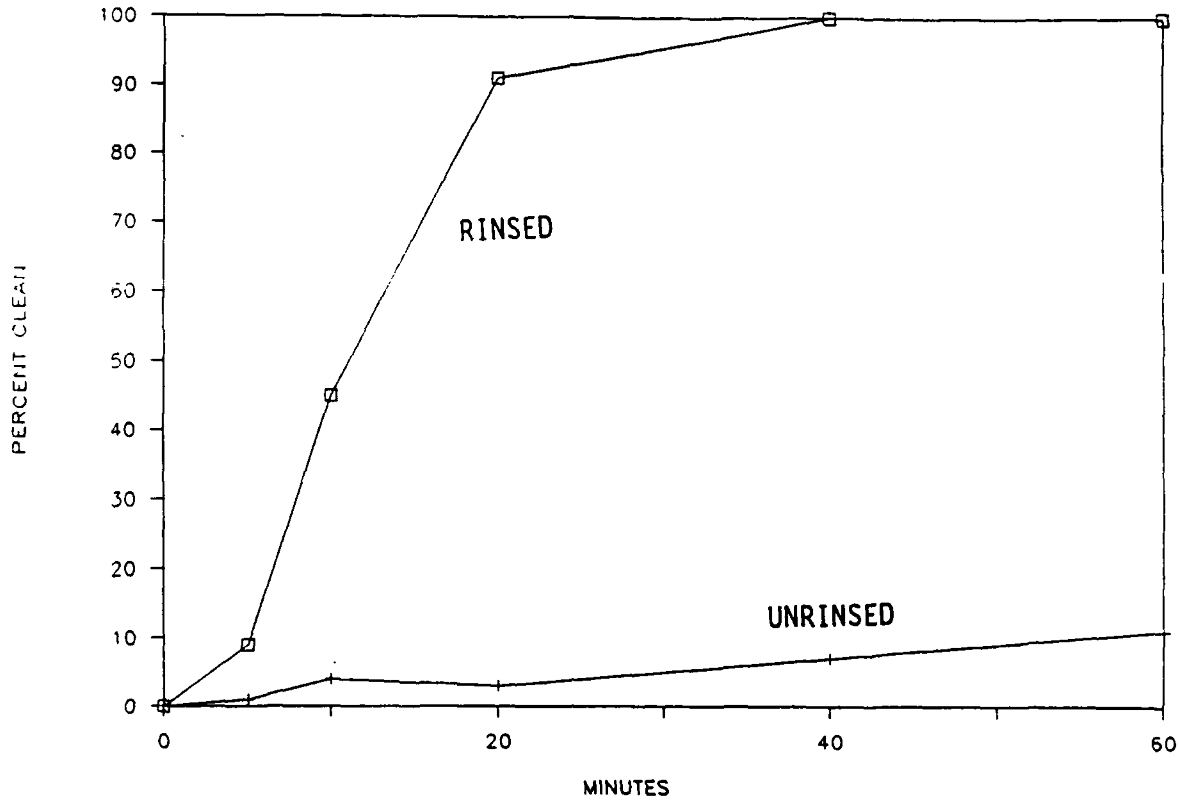

Figure 24. Cleaning Efficiency of Pacific Chemical, Riddall, on Oil/Xylene, 1:4, at Ambient Temperature.

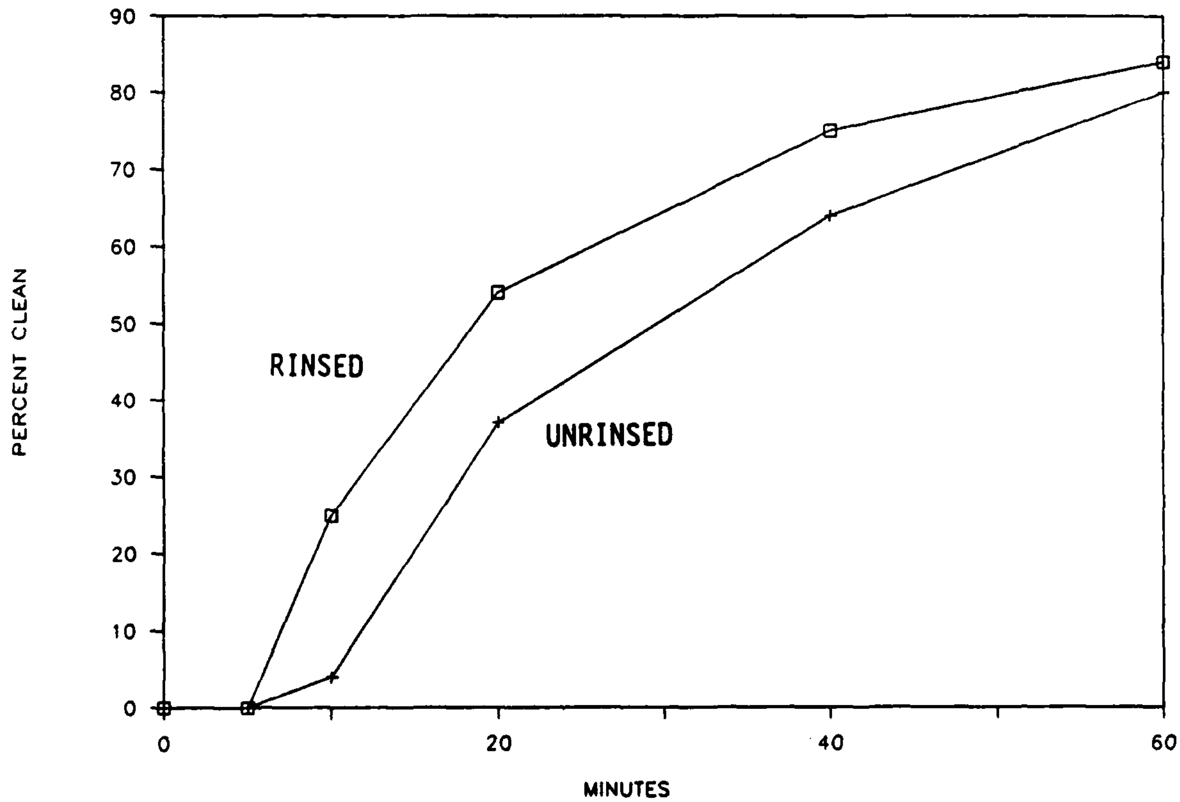

Figure 25. Cleaning Efficiency at Rochester Midland SE377C on Wax, 1:2, at 140°F.

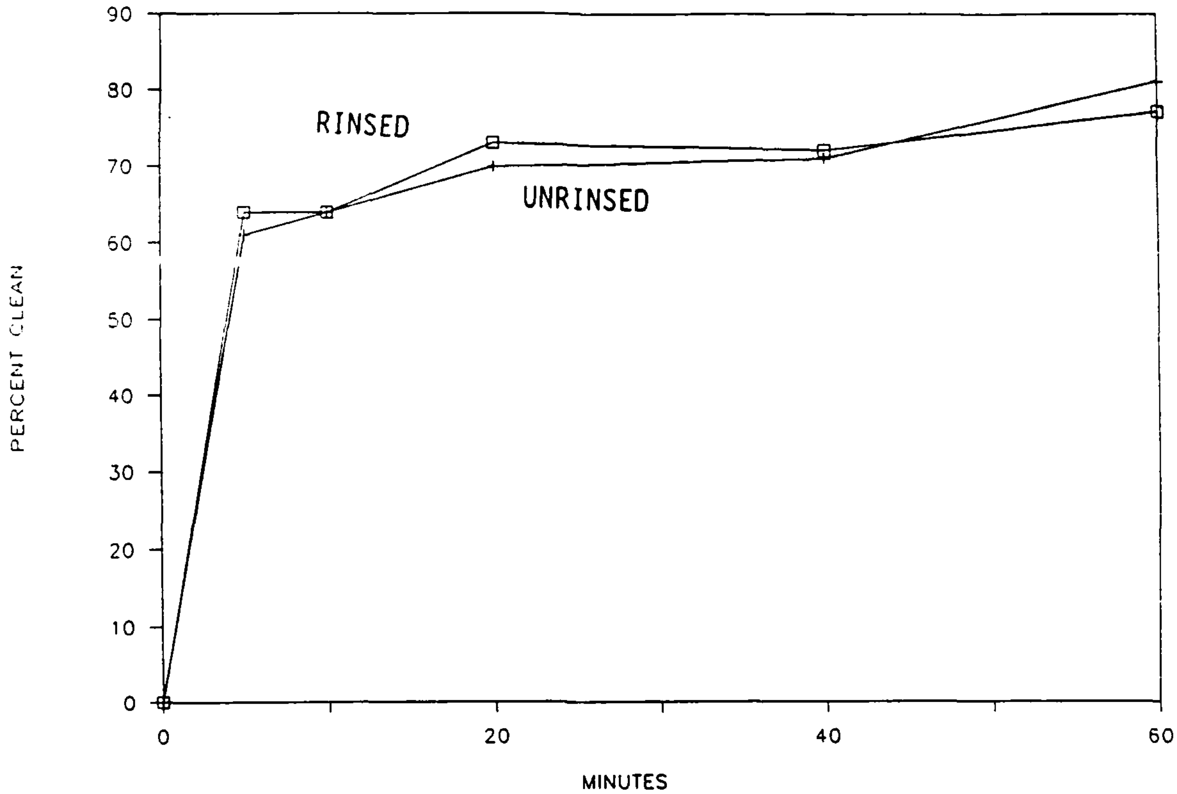

Figure 26. Cleaning Efficiency of Exxon 600 at 100°F.

Rochester Midland SE377C at 140°F, wax in Exxon Exxate 600 at 100°F, and Zep Big Orange at ambient temperature after rinse. One possible explanation for these differing curves is differing "reactions" of the soil with the solvent. In Figure 24 there is solubilization or softening as a function of time as the solvent penetrates to the metal surface; however, the soil is removed by flaking off and does not dissolve into the solvent. This is indicated, too, by the increased wax removal with rinsing under running water after dipping in the solvent (Figure 24) and the observation that in some cases the soil was removed as flakes either into the solvent or when rinsing.

Figure 25, Rochester Midland SE277C, represents a surface phenomenon, or first order kinetics. The third curve may indicate a bond or reaction between the metal and the soil as with grease in the Exxon Exxate 600 at 100°F (Figure 26) or repulsion between the soil and solvent. In these cases, increasing the soil exposure to the solvent does not increase the removal efficiency, nor does rinsing increase soil removal.

Rinsing as an enhancement method was evaluated. In many cases, cleaning efficiency increased from 0-10% with no rinsing to nearly 100% with rinsing as in Figure 24. However, in other cases, as in Figures 26, rinsing did not increase cleaning efficiency.

C. BIODEGRADABILITY

To establish a basis for comparison, the biodegradability tests of the solvents were run with appropriate controls and standards. Phenol was used as the standard solvent, because it is the solvent currently treated at Tinker AFB IWTP. Changes in biomass, biological activity, and biodegradation were compared to the controls in the test columns, which contained phenol. Examination of the data indicated the following activities.

Acclimation of the organisms to certain solvents tested was apparent. This acclimation was evidenced by a delay of biodegradation activity for 2-3 hours followed by a period of sustained degradation (Figure 27).

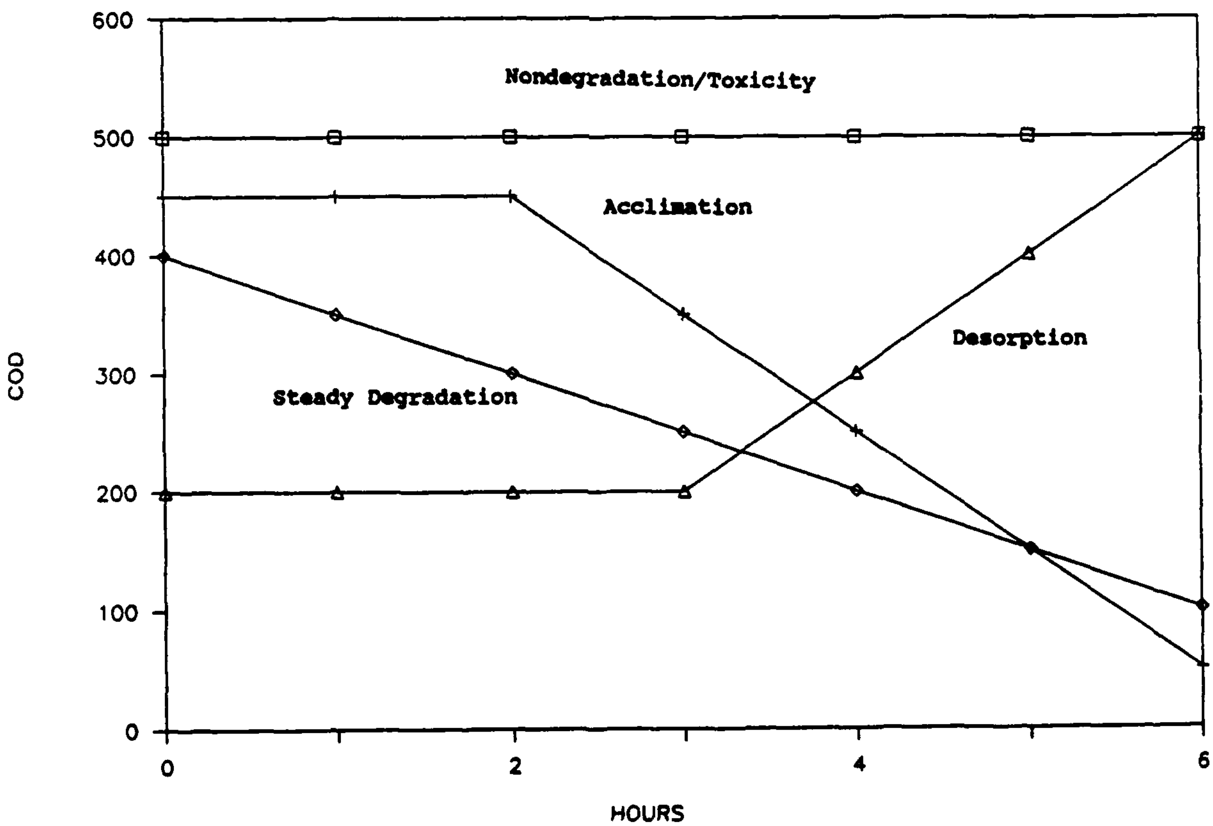

Figure 27. Examples of Biodegradation in the Biodegradability Test.

Another condition that became evident was toxicity or recalcitrance of certain solvents. This was indicated by no loss of COD during the entire testing period, which showed that the material was resistant to biological degradation during the contact time permitted. ATP data were used to determine if the solvent was toxic to the microorganisms. A severe decrease of ATP over the test period indicated that the material was particularly toxic to the activated sludge used as seed in the test columns (Figure 27). A minor decrease in ATP was expected to occur in test solvents that were resistant to degradation, but were not toxic. Additional testing was necessary to distinguish between actual toxicity and recalcitrance in areas of possible overlap of these conditions.

Air stripping and sorption of certain solvents was also evident (Figure 27). Large losses in CODs over the testing period may represent loss due to biological activity, sorption of the solvent to the biomass or container walls, or solvent volatility. Again, the ATP data were used to determine if the loss of solvent indicated increased biological activity. High final ATP levels indicated that the biomass was active during the testing period, thus supportive of biodegradability. Low ATP levels were indicative of possible stripping. In either case, these solvents were tested for air stripping by submitting them to the same testing conditions without inoculation of the microorganisms. COD losses measured during these tests were assumed to be either sorption to the container walls or loss due to volatility.

Desorption (Figure 27) is exhibited by an increase in COD values during the testing period. The solvent may adsorb to the biomass and container when first added to the test column. As the material is released from the container or biomass, measured COD levels may increase if the material is not biodegradable to any great extent.

Although these conditions are apparent in the results accumulated to date, insufficient data were collected to correctly identify some of these activities and conditions without further testing. Air stripping and sorption tests were conducted to elucidate a few of the test results.

In the tests of solvents for grease and hydraulic fluid removal, a number of solvents met the cleaning and biodegradability requirements. To decrease the number of corrosion tests and solvents, the criteria of temperatures of 140°F or less and solvents applicability to all metals were added. Only two solvents, 3D Supreme (1:3 at 100°F) and Calla 301 (1:1 at 100°F), required further testing.

D. CORROSION

The solvents which were biodegradable and which cleaned wax, oil/xylene, grease, or hydraulic fluid with greater than 80% efficiency were selected for corrosion testing (Table 5). Each of these solvents were tested on the metals listed in Table 1.

In general, the solvents had little effect on the metals after exposure for 168 hours at temperature. No pitting was evident on any of the metals. Appendix H contains the results of the corrosion tests.

TABLE 5. CORROSION TEST CONDITIONS

Company/Solvent	Temperature (°F)	Concentration
3D Supreme	100	1:1
Calla Chemical Calla 301	100	1:3
Bio-Tek Safety Solvent	150	Concentrate
Bio-Tek 134 Hi-Sol	150	Concentrate
Crowley MethylNaphtalene #5	120	Concentrate
Exxon Exxate 600	140	Concentrate
Exxon Exxate 700	120	Concentrate
Exxon Exxate 800	140	Concentrate
Exxon Exxate 900	140	Concentrate
Exxon Exxate 1000	140	Concentrate
Exxon Exxate 1300	140	Concentrate
Exxon Exxate 1000/Norpar 13	120	Concentrate
Exxon Norpar 13	140	Concentrate
Exxon Norpar 15	120	Concentrate
Exxon WS2443	120	Concentrate
Exxon Exxsol D80	120	Concentrate
Exxon Exxsol D110	120	Concentrate
Exxon Aromatic 150	120	Concentrate
Orange Sol De-Solv-It	140	Concentrate
Triton Hemo-Sol	100	Concentrate

Triton Hemo-Sol caused surface oxidation of the copper (CDA110) and the admiralty brass (CDA443). The average corrosion rate was 0.18 mil/yr for copper and 0.14 mil/yr for admiralty brass. This is less than the criterion of 0.3 mil/yr established for 1,1,1-trichloroethane. Carbon steel C4340 with Hemo-Sol had a corrosion rate of 0.38 and 0.43 mil/yr for two of the coupons; the third coupon had a rate of 0.06 mil/yr. The C1020 carbon steel coupons had an average corrosion rate of 0.06 mil/yr; the rates ranged from 0.02-0.13 mil/yr in the same solvent.

In Crowley Methylnaphthalene 5, aluminum Al7075, Monel MK-500, titanium, and carbon steel C1020 had some discoloration, but the corrosion rate was negligible (1.0-0.03 mil/yr).

After exposure to the Orange Sol De-Solv-It, carbon steel C4340 had a light brown discoloration as did Monel MK-500 and Stainless Steel-410. Again, the corrosion rate was negligible.

Bio-Tek 134 Hi-Solv caused some discoloration or rusting on both carbon steels C1020 and C4340. However, the corrosion rate was less than 0.3 mil/yr. 3D Supreme and Calla 301 caused oxidation of the copper CDA110 and Admiralty Brass CDA443. The average corrosion rate for the 3D Supreme at 1:3 concentration was 0.32 and 0.89 mil/yr, respectively. Exposure to the concentrate increased the corrosion rate 0.69 and 1.12 mil/yr. No pitting or localized attack was observed on the metals. Calla 301 showed a corrosion rate of 0.19 and 0.35 mil/yr in the 1:1 solution.

Those solvents showing surface effects, significant corrosion rates, or discoloration will require further corrosion testing to determine the effect of the solvent over an extended period. Additionally, all the solvents should be tested for hydrogen embrittlement effects.

SECTION V
CONCLUSIONS

With the procedures developed for screening solvents in this project, solvents can be quickly and accurately screened for application and biodegradability for Air Force processes. The method developed for biodegradation testing will be submitted as an ASTM standard method.

As evidenced by initial screening results, solvents are available that are technically feasible for replacing chlorinated solvents used for masking-wax removal. These solvents are biodegradable as specified by the criteria established in this report. The solvents which meet all these criteria are listed in Table 6.

Cleaning tests indicated that for several of the solvents, process enhancements such as stirring or rinsing the coupons increased cleaning efficiency, and cleaned the coupons at lower temperatures.

Corrosion tests indicate that for the most part, the solvents passed the corrosion criterion of less than 0.3 mil/yr, but some of them did show signs of surface oxidation or corrosion. These solvents require additional testing.

Biodegradability tests indicated that for some of the solvents, the organisms required acclimation to the solvent. Some of the solvents were air stripped by the system rather than being degraded by the organisms. In some cases, the solvents were toxic to the organisms with no change in degradation with time. With time, there was also evidence of desorption of the solvents from the organism.

TABLE 6. SOLVENTS PASSING CLEANING AND BIODEGRADATION TESTS

3D	SUPREME	GREASE/HYDRAULIC FLUID
BIO-TEK INC.	134 HI-SOLV	HYDRAULIC FLUID/WAX
BIO-TEK INC.	SAFTEY SOLVENT DEGREASER	HYDRAULIC FLUID/OIL-XYLENE/WAX/GREASE
BRULIN	CL-500	GREASE
CALLA CHEMICAL CORP	CALLA 301	GREASE
CROWLEY CHEMICAL COMPANY	METHYL NAPHTHALENE #5	HYDRAULIC FLUID/OIL-XYLENE/WAX/GREASE
DOBER CHEMICAL CORP.	8679	HYDRAULIC FLUID/GREASE
ELGENE CHEMICALS INC.	FABULENE	HYDRAULIC FLUID/GREASE
ELGENE CHEMICALS INC.	#22 SKIDOO	HYDRAULIC FLUID/GREASE
EXXON CHEMICALS	EXXATE 1000	HYDRAULIC FLUID/WAX/OIL-XYLENE
EXXON CHEMICALS	EXXATE 1300	HYDRAULIC FLUID/WAX/OIL-XYLENE
EXXON CHEMICALS	EXXATE 600	HYDRAULIC FLUID/WAX/GREASE
EXXON CHEMICALS	EXXATE 800	HYDRAULIC FLUID/WAX/OIL-XYLENE
EXXON CHEMICALS	EXXATE 900	HYDRAULIC FLUID/OIL-XYLENE
FREDERICK GUMM CHEM.	CLEPO 136-R	GREASE
GRAYMILLS CORP.	AQUATENE 571	GREASE
GRAYMILLS CORP.	AQUATENE 581	HYDRAULIC FLUID/WAX/GREASE
HUNTINGTON	LHTS	GREASE
INTERNATIONAL PRODUCTS CO	MICRO	HYDRAULIC FLUID/GREASE
KIESOW INTERNATIONAL CORP	EKASIT E-231	HYDRAULIC FLUID/GREASE
KIESOW INTERNATIONAL CORP	EKASIT S-261	HYDRAULIC FLUID/GREASE
MAGNUSON PRODUCTS	PERMAG #404	HYDRAULIC FLUID/GREASE
MITCHELL-BRADFORD, INTERN	MI-CLEAN 100	HYDRAULIC FLUID/GREASE
MITCHELL-BRADFORD, INTERN	MI-CLEAN 14	HYDRAULIC FLUID/GREASE
MITCHELL-BRADFORD, INTERN	MI-CLEAN 31	GREASE
MITCHELL-BRADFORD, INTERN	MI-CLEAN 59	HYDRAULIC FLUID/GREASE
OAKITE PRODUCTS INC.	OAKITE 162	HYDRAULIC FLUID/GREASE
OAKITE PRODUCTS INC.	OAKITE 2327	HYDRAULIC FLUID/GREASE
OAKITE PRODUCTS INC.	OAKITE 24	HYDRAULIC FLUID/GREASE
OAKITE PRODUCTS INC.	OAKITE ANOPREP	HYDRAULIC FLUID/GREASE
OAKITE PRODUCTS INC.	OAKITE HD 126	HYDRAULIC FLUID/GREASE
ORANGE-SOL INCORPERATED	DE-SOLV-IT	HYDRAULIC FLUID/GREASE/WAX/OIL-XYLENE
PANTHER CHEMICAL CO.	RR 50	HYDRAULIC FLUID/GREASE
PARKER CHEMICAL	PARCO CLEANER 2033	HYDRAULIC FLUID/GREASE
PARKER CHEMICAL	PARCO CLEANER 2053	HYDRAULIC FLUID/GREASE
PATCLIN CHEMICAL CO.	#309	HYDRAULIC FLUID/GREASE
PATCLIN CHEMICAL CO.	#348	HYDRAULIC FLUID/GREASE
PROGRESS CHEMICAL INC.	AC-1	HYDRAULIC FLUID/GREASE
TRI TON	HEMO-SOL	WAX

SECTION VI
RECOMMENDATIONS

The results of the work to date indicate that solvents are available that can remove wax and grease efficiently and can be treated in the IWTP system. However, tests also indicate that the solvents may require enhancement processes and acclimation of the organisms to the solvents for complete degradation. The results do not provide information on the life of the solvents or the rinsing requirements. It is recommended that the solvents be tested to provide these data. Included in the test should be (a) extended biodegradability testing in the pilot-scale activated sludge treatment system at Tinker AFB, (b) cleaning tests with actual aircraft parts to determine the solvents' life and rinsing requirements, and (c) extended corrosion testing. The test plan for these tests are presented below.

A. PHASE II TEST PLAN

The solvents that passed the initial screening tests for biodegradability will be tested for cleaning efficiency, corrosion, and the effects on the IWTP to develop the economics of the replacement solvents. Tasks will include:

- Solvent Performance Testing
- Bioacclimation Tests
- Enhancement Method Testing
- Extended Corrosion Testing
- Human Toxicity Data Gathering
- Sensor Development
- ASTM Method Development

The solvents selected for these tests are Bio-Tek Safety Solvent, Exxon Exxate 600, Orange-Sol Desolv-It, Triton Hemo-Sol, 3D Supreme, and Calla 301.

1. Solvent Performance Testing

To define the economics of the replacement solvents, we must establish the life expectancies, rinse water requirements, and drying equipment that may be required. Reject parts will be coated with masking wax and cleaned in dip tanks with the replacement solvents at our testing facility. Then if the part requires rinsing, it will be rinsed to determine how much and what type of rinsing is required (spray, dip, etc.). After rinsing, the part will be allowed to dry to determine if blowers are required to remove the residual liquid. This process will be repeated while testing the solvent to ensure that it still meets the quality requirements and that it has completely cleaned the part. Once the solvent has been exhausted, it will be fed to the pilot-scale IWTP in concentrations that the actual IWTP would expect. These tests will be repeated for the duration of the bioacclimation tests.

2. Bioacclimation

To establish the effects of the replacement solvents on the IWTP's activated sludge treatment system, we will track the parameters listed in Table 7. The duration of the test will depend on the activated sludge basin's turnover. Approximately three sludge ages are required to obtain a stable basin following the introduction of a new compound. This is expected to take from 15-21 days. following a test with a replacement solvent, three sludge ages will be required for the system to return to normal operating conditions.

During each test we will use on-line respirometry to evaluate toxicity and acclimation for determining if the activated sludge is accepting or rejecting the new solvent. Spike additions will be used to determine through-line acclimation for the long-term tests. The on-line respirometer will also be used to identify any potential changes in the activated sludge system's operating conditions (i.e., nutrients, pH).

TABLE 7. ACTIVATED SLUDGE PARAMETERS COLLECTED DAILY

Mixed Liquor Suspended Solids (MLSS) in Aeration Tank (mg/L)
Volatile Suspended Solids (VSS) in Aeration Tank (mg/L)
Volatile Suspended Solids in Waste Sludge (VSS_{ws}) (mg/L)
Suspended Solids in Waste Sludge (SS_{ws}) (mg/L)
Influent Suspended Solids (SS_i) (mg/L)
Influent Wastewater Biochemical Oxygen Demand (BOD_i) (mg/L)
Effluent Wastewater Biochemical Oxygen Demand (BOD_e) (mg/L)
Aeration Tank Volume (V) (ft^3)
Settled Waste Water Flow (Q) (gpm)
Return Sludge Flow (RSF) (gpm)
Waste Sludge Flow (WSF) (gpm)
Total Organic Carbon (TOC) (mg/L)
Chemical Oxygen Demand (COD) (mg/L)

3. Enhancement Methods

During Phase II we will evaluate several methods of enhancement to increase the efficiency of the solvents or decrease the required operating temperatures. The methods investigated will include mixer agitation and ultrasonic agitation. Screening test similar to those of Phase I will be conducted. Coupons will be completely coated with the soil and cleaned in 5-gallon ultrasonic tanks using the different enhancement methods. For each enhancement method, the percent efficiency, the energy input, and the impact on solvent life will be evaluated.

4. Extended Corrosion Testing

During Phase II, extended corrosion tests will be run to determine several data points that were not found during the preliminary tests run in Phase I. These will include tests to determine the hydrogen embrittlement effects of the replacement solvents. Also during this phase, we will run longer-term corrosion tests on several of the solvents that showed corrosive action on the alloys in the short-term tests. We will also run tests to determine if the enhancement methods increase the corrosiveness of the solvent.

5. Human Toxicity Data Gathering

Replacing chlorinated solvents with substances that are biodegradable but potentially more hazardous to humans would not solve the present problem facing the Air Force. To avoid such a situation, existing data will be collected and compiled on the solvents tested in Phase II. For those solvents which human toxicity data is not available, manufacturers will be encouraged to have the tests run by independent laboratories.

6. Sensor Development

The need for another important task in Phase II was discovered after extensive evaluations of the processes used at the ALCs. Presently, the cleaning tanks are monitored weekly by technicians who measure the important active ingredients in each tank. With the new biodegradable solvents, this may require much more monitoring. Such solvents may require higher maintenance and turnover, requiring technicians to analyze the solution several times a week or possibly daily. Such extensive monitoring would greatly increase the operating costs associated with these new solvents.

To reduce these operating costs, an on-line, low-maintenance sensor must be developed to monitor solutions continuously and help maintain optimum operating parameters. Recent research at the INEL has shown that ultrasonic sensors can measure concentrations of different chemical species in a solution by measuring the speed of sound. Different chemical species affect the speed of sound to different degrees, depending on their density and the strength of the intermolecular forces present in the medium. It has been shown that this method can measure changes in concentrations of a solute to less than one part per million and distinguish between several different solutes. Adapting ultrasonic technology to this application will require some developmental work.

7. ASTM Method Development

Another important part of Phase II is the development of an ASTM standard for "what is biodegradable." During initial planning, it was learned that there is presently no standard method for determining if a solvent is biodegradable. Such a method was needed for solvent screening, so we developed a method using an activated sludge system to degrade the solvents. This method is explained in Section III E.

An extensive write-up of the proposed method was presented at the spring meeting of the Biological Effects and Fate Chapter of the ASTM in April 1988. The committee was extremely receptive to the need for such a standard, and the informal review resulted in a request for a formal presentation of the proposed standard method. This proposal will be prepared in Phase II.

Several additional steps must be undertaken during Phase II for this method to be validated as an acceptable method. These include:

- Verification of assumptions
- Tracking of the major components
- Final fate of the major components
- Toxicology of the solvent on the biological culture
- Adaptation of the culture

Development of such a standard is extremely important to this program for it to succeed. For these biodegradable solvents to be accepted for use by the Air Force, they must be integrated into T.O.s and Mil Specs. A method that any technical person can follow is a must. One of the advantages of an ASTM standard is that it is accepted as an extensively peer reviewed and accepted procedure that is accurate and reproducible.

An ASTM method for defining biodegradability will be invaluable in evaluating the effects, on an IWTP, of replacing any compound in a process facility. If phenol is replaced in paint stripping tanks, it can be determined quickly, easily, and accurately if the new stripper can be treated in the activated sludge system.

From the results of this testing, solvents should be selected for demonstration in an actual process, and the specifications established for future biodegradable solvent selection.

REFERENCES

1. ANSI/ASTM F 483-77, Standard Method of Total Immersion Corrosion test for Aircraft Maintenance Chemicals.

2. Biodegradability of Alkylbenzene Sulfonates (Designation: D2667-82, Presumptive Test), April 1982.

3. McCoy, J.W., Industrial Chemical Cleaning, Chemical Publishing Co., New York, 1982.

4. T.O. 42C2-1-7, Process Instructions, Metal Surface Treatments to Meet Air Force Requirements, March 1979.

5. MIL-G-21164, Grease Molybdenum Disulfide for Low and High Temperatures, NATO Code No. G-353, December 8, 1981.

6. MIC-C-85570B, Cleaning Compound, Aircraft Exterior, December 1986.

7. MIL-H-83282, Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Aircraft, NATO Code No. H537.

8. MIL-C-43616B, Purchase Description, Remover, Activated Solvent Type, for Difficult-to-Remove Finishes for SA-ALC and OC-ALC Directorate of Maintenance, February 19, 1985.

9. "Basic EPA Nutrient Media Reference," Federal Register, Vol. 50, No. 188, p. 39279, September 27, 1985.